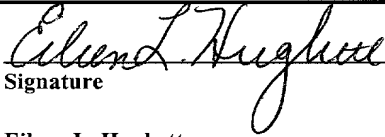


410 Rec'd PCT/PTO 06 APR 2000

FORM PTO-1390 OFFICE (REV 10-95)		U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK	ATTORNEY'S DOCKET NUMBER 6873 U.S. APPLICATION NO. (if known, see 37 CFR 1.5) 09/509603
TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371			
INTERNATIONAL APPLICATION NO. PCT/US98/20513	INTERNATIONAL FILING DATE 01 October 1998	PRIORITY DATE CLAIMED	
TITLE OF INVENTION DETERGENT COMPOSITION FOR HARD SURFACES COMPRISING HYDROPHILIC SHEAR-THINNING POLYMER AT VERY LOW LEVEL			
APPLICANT(S) FOR DO/EO/US POLICICCHIO, Nicola John; SHERRY, Alan Edward; ; ;			
Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information.			
<p>1. <input checked="" type="checkbox"/> This is a FIRST submission of items concerning a filing under 35 U.S.C. 371.</p> <p>2. <input type="checkbox"/> This is a SECOND or SUBSEQUENT submission of items concerning a filing under 35 U.S.C. 371.</p> <p>3. <input type="checkbox"/> This express request to begin national examination procedures (35 U.S.C. 371(f) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(I).</p> <p>4. <input checked="" type="checkbox"/> A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.</p> <p>5. <input checked="" type="checkbox"/> A copy of the International Application was filed (35 U.S.C. 371(c)(2))</p> <p> a. <input type="checkbox"/> is transmitted herewith (required only if not transmitted by the International Bureau).</p> <p> b. <input type="checkbox"/> has been transmitted by the International Bureau.</p> <p> c. <input checked="" type="checkbox"/> is not required, as the application was filed in the United States Receiving Office (RO/US).</p> <p>6. <input type="checkbox"/> A translation of the International Application into English (35 U.S.C. 371(c)(2)).</p> <p>7. <input checked="" type="checkbox"/> Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))</p> <p> a. <input type="checkbox"/> are transmitted herewith (required only if not transmitted by the International Bureau).</p> <p> b. <input type="checkbox"/> have been transmitted by the International Bureau.</p> <p> c. <input type="checkbox"/> have not been made; however, the time limit for making such amendments has NOT expired.</p> <p> d. <input checked="" type="checkbox"/> have not been made and will not be made.</p> <p>8. <input type="checkbox"/> A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).</p> <p>9. <input checked="" type="checkbox"/> An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).</p> <p>10. <input type="checkbox"/> A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).</p> <p>Items 11. to 16. below concern document(s) or information included:</p> <p>11. <input type="checkbox"/> An Information Disclosure Statement under 37 CFR 1.97 and 1.98.</p> <p>12. <input type="checkbox"/> An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.</p> <p>13. <input checked="" type="checkbox"/> A FIRST preliminary amendment.</p> <p> <input type="checkbox"/> A SECOND or SUBSEQUENT preliminary amendment.</p> <p>14. <input type="checkbox"/> A substitute specification.</p> <p>15. <input checked="" type="checkbox"/> A change of power of attorney and/or address letter.</p> <p>16. <input type="checkbox"/> Other items or information:</p> <p>"Express Mail" mailing label number <u>E6348982014US</u></p> <p>Date of Deposit <u>06 April 2000</u></p> <p>I hereby certify that this paper/fee is being deposited with the United States Postal Service "Express Mail Post Office to Addressee" service under 37 CFR 1.10 on the date indicated above and is addressed to The Assistant Commissioner of Patents, Washington, D.C. 20231</p> <p>Administrator Mailing Application <u>Samuel Butta</u> Signature</p>			

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U.S. APPLICATION NO. (if known, see 37 CFR 1.5) 09/509603		INTERNATIONAL APPLICATION NO. PCT/US98/20513		ATTORNEY'S DOCKET NUMBER 6873	
				CALCULATIONS PTO USE ONLY	
ENTER APPROPRIATE BASIC FEE AMOUNT =				\$670	
Surcharge of \$130.00 for furnishing the oath or declaration later than [] 20 [] 30 months from the earliest claimed priority date (37 CFR 1.492(e)).				\$0	
CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE		
Total Claims	28-20 =	8	x \$22.00	\$176	
Independent Claims	5-3 =	2	x \$78.00	\$156	
MULTIPLE DEPENDENT CLAIM(S) (if applicable)			\$250.00	\$0	
TOTAL OF ABOVE CALCULATIONS =				\$1002	
Processing fee of \$130.00 for furnishing the English translation later than [] 20 [] 30 months from the earliest claimed priority date (37 CFR 1.492(f)).				\$0	
TOTAL NATIONAL FEE =				\$1002	
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28,3.31). \$40.00 per property +				\$0	
TOTAL FEES ENCLOSED =				\$1002	
				Amount to be refunded	\$
				charged	\$
<p>a. [] A check in the amount of \$ ____ to cover the above fees is enclosed.</p> <p>b. [x] Please charge my Deposit Account No. <u>16-2480</u> in the amount of \$ <u>1002</u> to cover the above fees. A duplicate copy of this sheet is enclosed.</p> <p>c. [x] The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. <u>16-2480</u>. A duplicate copy of this sheet is enclosed.</p> <p>NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.</p> <p>SEND ALL CORRESPONDENCE TO:</p>					
J.J. Camp Patent Division The Procter & Gamble Company Sharon Woods Technical Center 11510 Reed Hartman Highway Cincinnati, OH 45241				 Signature Eileen L. Hughett Name 34,352 Registration Number	

Case 6873

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In the matter of :
:
U.S. National Phase Entry :
Under 35 USC 371 from :
the International Application of :
POLICICCHIO, Nicola John et al :
Int'l Application No. PCT/US98/20513 :
Filed in the RO/US on 01 October 1998 :
Entitled: Detergent Composition For Hard Surfaces :
Comprising Hydrophilic Shear-Thinning Polymer :
At Very Low Level :

PRELIMINARY AMENDMENT

Assistant Commissioner for Patents
Box PCT
Washington, D.C. 20231

Dear Sir:

Before computing the fees for entering the captioned International Application into the U.S. National Phase, please enter the following amendments **IN THE CLAIMS**:

- * Please cancel Claims 1 thru 10.

Please add the following new claims:

11. Detergent composition for cleaning floors comprising from about 0.0001% to about 0.2% of hydrophilic, shear-thinning polymer that is capable of inhibiting molecular aggregation of surfactant solution on floors during the dry-down process.
12. The detergent composition according to Claim 11 wherein the level of said polymer is from about 0.0001% to about 0.1%.
13. The detergent composition according to Claim 12 wherein the level of said polymer is from about 0.0005% to about 0.08%.
14. The detergent composition according to Claim 11 wherein the hydrophilic, shear-thinning polymer has a molecular weight of at least about 100,000.
15. The detergent composition according to Claim 14 wherein the hydrophilic, shear-thinning polymer has a molecular weight of at least about 1,000,000.

16. The detergent composition of Claim 11 wherein the hydrophilic polymer is selected from the group of polymers consisting of xanthan gum, guar gum, gum arabic, pectin and mixtures thereof.

17. The detergent composition of Claim 16 wherein the hydrophilic polymer is xanthan gum.

18. The detergent composition of Claim 11 wherein the level of detergent surfactant is from about 0.01% to about 0.50%; the level of one or more solvents is less than about 5.0%; and the pH is greater than about 9.

19. The detergent composition of Claim 18 wherein the level of detergent surfactant is from about 0.05% to about 0.30%; the level of one or more solvents is less than about 5.0%; and the pH is greater than about 9.5.

20. The detergent composition of Claim 19 wherein the level of detergent surfactant is from about 0.10% to about 0.20%; the level of one or more solvents is less than about 5.0%; and the pH is greater than about 10.0.

21. A detergent composition according to Claim 20 wherein the composition is obtained by dilution with from about 1:50 to about 1:250 parts per part of the composition of water of a more concentrated product form prior to use.

22. The detergent composition of Claim 21 containing an effective amount of suds suppressor.

23. The detergent composition of Claim 22 wherein said suds suppressor is at a level of from about 0.0005% to about 0.02%.

24. The detergent composition of Claim 23 wherein said suds suppressor is at a level of from about 0.001% to about 0.01%.

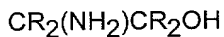
25. The detergent composition of Claim 24 wherein said suds suppressor comprises a silicone suds suppressor.

26. The detergent composition of Claim 11 wherein the detergent surfactant has a predominantly linear structure.

27. The detergent composition of Claim 26 wherein the detergent surfactant has a linear structure and is selected from the group consisting of straight chain anionic and nonionic detergent surfactants.

28. The detergent composition of Claim 11 wherein the alkalinity is provided, at least in an effective amount, by volatile alkaline agents.

29. The detergent composition of Claim 28 wherein the volatile alkaline agent is an alkanol amine having the formula:



wherein each R is selected from the group consisting of hydrogen and alkyl groups containing from one to four carbon atoms and the total of carbon atoms in the compound is from three to six.

30. The detergent composition of Claim 29 wherein the volatile alkaline agent is 2-amino-2-methyl-1-propanol.

31. A kit comprising an implement containing a pad containing superabsorbent material and a detergent composition containing no more than about 0.5% detergent surfactant; a level of one, or more solvents, that is less than about 5.0%; and a pH of more than about 9.

32. The detergent composition of Claim 11 in a container, in association with instructions to use it with a pad containing a superabsorbent material.

33. The process of cleaning a surface comprising applying an effective amount of a detergent composition containing no more than about 0.5% detergent surfactant; a level of one or more solvents, that is less than about 5.0%; and a pH of more than about 9 and absorbing the composition in an absorbent structure comprising a superabsorbent material.

34. The process of Claim 33 wherein said surface is ceramic and said detergent composition comprises an effective amount of suds suppressor.

35. The process of Claim 34 wherein said surface is vinyl or a polyurethane coated vinyl surface and said detergent composition comprises an effective amount of suds suppressor.

36. The process of Claim 35 wherein said surface is wood and pre-finished wood and wood laminates and said detergent composition comprises an effective amount of suds suppressor.

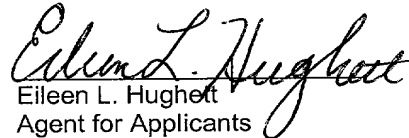
37. The process of cleaning a surface comprising applying an effective amount of a detergent composition containing no more than about 0.5% detergent surfactant; a level of one or more solvents, that is less than about 5.0%; and a pH of more than about 9 and absorbing the composition using a string, sponge, strip mop or floor cloth.

38. The process of cleaning a surface comprising applying an effective amount of a detergent composition containing no more than about 0.5% detergent surfactant; a level of one or more solvents, that is less than about 5.0%; and a pH of more than about 9 and absorbing the composition in an absorbent structure comprising a superabsorbent material.

The support for these amendments is found in the claims as originally filed. These amendments are being entered to bring the claims into conformance with, *inter alia*, 37 CFR §1.75; no new matter is added.

Respectfully submitted for Applicants,

By:


Eileen L. Hughett
Agent for Applicants
Registration No. 34,352

04 April 2000
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PCT Rec'd 06 APR 2000

**DETERGENT COMPOSITION FOR HARD SURFACES COMPRISING
HYDROPHILIC SHEAR-THINNING POLYMER AT VERY LOW LEVEL**

TECHNICAL FIELD

This application relates to detergent compositions that can be used for hard surfaces, and especially for floor cleaning, including conventional applications and implements such as sponges, cloths, sponge mops, string mops, strip mops and floor cloths. This application is also especially useful with a "disposable" cleaning implement comprising a superabsorbent material for removing soils from hard surfaces. The cleaning implements preferably comprise a removable absorbent cleaning pad, preferably designed so as to provide multiple cleaning surfaces.

BACKGROUND OF THE INVENTION

The literature is replete with products capable of cleaning hard surfaces such as ceramic tile floors, hardwood floors, counter tops and the like. In the context of cleaning floors, numerous devices are described comprising a handle and some means for absorbing a fluid cleaning composition. Such devices include those that are reusable, including mops containing cotton strings, cellulose and/or synthetic strips, sponges, and the like. The use of any such device or mop requires considerable effort.

Examples of disposable mops include: U.S. Patent No. 5,094,559, issued March 10, 1992 to Rivera et al., which describes a mop that includes a disposable cleaning pad comprising a scrubber layer for removing soil from a soiled surface, a blotter layer to absorb fluid after the cleaning process, and a liquid impervious layer positioned between the scrubber and blotter layer and U.S. Patent 5,419,015, issued May 30, 1995 to Garcia, which describes a mop having removable, washable work pads, said patents being incorporated herein by reference.

The cleaning implement herein preferably comprises a removable cleaning pad, which alleviates the need to rinse the pad during use. This cleaning pad preferably possesses sufficient absorbent capacity, on a gram of absorbed fluid per gram of cleaning pad basis, to allow the cleaning of a large area, such as that of the typical hard surface floor (e.g., 80-100 ft²), without the need to change the pad. This typically requires the use of a superabsorbent material, preferably of the type disclosed hereinafter. The detergent composition that is used with such superabsorbent materials must be carefully formulated to avoid defeating the goal of using such superabsorbent material.

The preferred cleaning implements have a pad which offers beneficial soil removal properties due to continuously providing a fresh surface, and/or edge to contact

the soiled surface, e.g., by providing a plurality of surfaces that contact the soiled surface during the cleaning operation.

SUMMARY OF THE INVENTION

Detergent compositions used for cleaning hard surfaces such as floors, either full strength, or diluted, will normally contain sufficient detergent ingredients such as surfactant, builder, solvent etc., to enable the solution to provide excellent end result cleaning without causing build-up or stickiness. End use is based on how the product is intended for use; diluted, such as in case of floor cleaners and multi-purpose cleaners, or neat, such as in the case of sprays out of a bottle or sprays out of a mopping implement that is used with disposable or re-usable pads.

Typically the "end use" cleaning solution, either full strength, or diluted, contains less than about 0.5% by weight of the solution of detergent surfactant. The level of detergent surfactant in the end use cleaning solution is preferably from about 0.01% to about 0.5 %, more preferably from about 0.05% to about 0.4%, and even more preferably from about 0.05% to about 0.3%, by weight of the composition/cleaning solution. To aid in cleaning, one or more cleaning solvents, preferably hydrophobic cleaning solvents, can also be present. The level of solvent(s), when present, in the end use cleaning solution is preferably from about 0.1% to about 5.0%, more preferably from about 0.25% to about 4.0%, and even more preferably from about 0.5% to about 3.0%, by weight of the composition/cleaning solution.

To aid in cleaning when using conventional implements, e.g., cloths, sponges, and mops such as sponge, strip, or string, and to avoid hindering absorption when using with a pad containing superabsorbent materials, the pH is preferably more than about 9, more preferably more than about 9.5, and even more preferably more than about 10. The alkalinity should preferably be provided, at least in part, by volatile materials, to avoid streaking/filming problems.

For the purpose of helping to level the solution during drying the composition should contain a polymer that has hydrophilic and shear-thinning characteristics that is capable of inhibiting molecular aggregation of surfactant solution on floors during the dry-down process to provide one, or more, of the benefits of: strippability; avoidance of build-up; easy spreading of solution on hard surfaces such as floors; and maintaining a sufficient amount of water on the surface to level the ingredients remaining on the surface. By leveling we mean minimizing solution de-wetting from the surface during drying which, in turn, minimizes streaking. Because of this benefit, the polymer allows formulation at even low surfactant levels and allows for addition of solvents to aid in

cleaning without hurting filming/streaking. Overall this can also lead to less residue and floor stickiness.

The essential polymer herein is preferably present at only a very low level, that is from about 0.0001% to about 0.2%, preferably from about 0.0001% to about 0.1%, more preferably from about 0.0005% to about 0.08%, by weight of the cleaning solution. The level in product will reflect the type of use, full strength, or dilute. The polymer is preferably selected from the group consisting of; natural gums, especially xanthan gums, guar gums, gum arabic, and/or pectin; synthetic polymers such as poly(styrene sulfonate); poly(vinyl pyrrolidone); and mixtures thereof, as monomers and/or polymers. The most preferred is xanthan gum.

The detergent surfactant is preferably predominantly linear, e.g., aromatic groups should not be present, and the detergent surfactant is preferably relatively water soluble, e.g., having a hydrophobic chain containing from about 8 to about 14, preferably from about 8 to about 12, carbon atoms, and, for nonionic detergent surfactants, having an HLB of from about 9 to about 14, preferably from about 10 to about 13, more preferably from about 10 to about 12.

The composition can be used in the context of conventional hard surface, e.g., floor or multi-purpose cleaners and with conventional cleaning and/or mopping systems known in the art such as sponges and cloths, e.g., sponge mops, strip mops, string mops and floor cloths. Additionally, a preferred aspect the present invention relates to the use of the cleaning solutions/compositions with an all-in-one implement plus cleaning pad system. The cleaning pad preferably contains a suberabsorbent material and works synergistically with the described detergent composition/solution to provide better end result cleaning with greater convenience. This cleaning system is typically comprised of:

- a. a handle; and
- b. a removable cleaning pad comprising a suberabsorbent material and having a plurality of substantially planar surfaces, wherein each of the substantially planar surfaces contacts the surface being cleaned, and preferably a pad structure which has both a first layer and a second layer, wherein the first layer is located between the scrubbing layer and the second layer and has a smaller width than the second layer.

Depending on the means used for attaching the cleaning pad to the cleaning implement's handle, it may be preferable for the cleaning pad to further comprise a distinct attachment layer. In these embodiments, the absorbent layer would be positioned between the scrubbing layer and the attachment layer.

The detergent composition and, preferably, the implement of the present invention are compatible with all hard surface substrates, including wood, vinyl, linoleum, no wax floors, ceramic, Formica[®], porcelain, glass, wall board, and the like. The implement and detergent composition provide ease of cleaning, especially when the polymer is present to provide easier mopping and better results.

BRIEF DESCRIPTION OF THE DRAWING

Figure 1 is a perspective view of a cleaning implement used in the preferred embodiment which has an on-board fluid dispensing device which will dispense the detergent composition.

Figure 1a is a perspective view of a cleaning implement used in the preferred embodiment which does not have an on-board fluid dispensing device, so that the composition is supplied separately.

Figure 1b is a side view of the handle grip of the implement shown in Figure 1a.

Figure 2 is a perspective view of a removable cleaning pad of the implement.

Figure 3 is a perspective view of an absorbent layer of a disposable cleaning pad used in the preferred embodiment.

Figure 4 is a blown perspective view of the absorbent layer of a removable cleaning pad used in the preferred embodiment.

Figure 5 is a cross sectional view of a cleaning pad used in the preferred embodiment, taken along the y-z plane.

DETAILED DESCRIPTION

I. Detergent Composition

The detergent composition acts as a cleaning solution either when used full strength, or when diluted. The level of ingredients, therefore, have to be considered in the context of the end use. The essential polymer is only used at very low levels in the cleaning solution. Therefore, any concentrated composition should be packaged in association with instructions to dilute to the proper level.

The Polymer

As discussed hereinbefore, the level of polymer should be low, e.g., that is from about 0.0001% to about 0.2 %, preferably from about 0.0001% to about 0.1% more preferably from about 0.0005% to about 0.08%, by weight of the composition. This very low level is all that is required to produce a better end result cleaning and higher levels can cause streaking/filming, build up, and/or stickiness.

While not wishing to be limited by theory, two physical properties are considered critical for the polymer: 1) Hydrophilic nature and 2) Shear-thinning ability. The polymer hydrophilicity is important to ensure strippability in-between cleanings to avoid

build-up. The shear-thinning characteristic is important in aiding to spread solution out evenly during use and combined with hydrophilic characteristic helps provide leveling effect. By leveling effect we mean minimizing solution de-wetting and molecular aggregation which typically occurs during dry down.. Molecular aggregation leads to visual streaking/filming which is a signal of poor end result cleaning.

Suitable examples of polymers include cellulose materials, e.g., carboxymethylcellulose, hydroxymethylcellulose, etc., and synthetic hydrophilic polymers such as polystyrene sulfonate. More preferred are naturally occurring polymers like gum arabic, pectin, guar gum and xanthan gum. Xanthan gum is particularly preferred. Xanthan gum is disclosed in U.S. Pat. No. 4,788,006, Bolich, issued Nov. 29, 1986, at Col. 5, line 55 through Col. 6, line 2, said patent being incorporated herein by reference. Many synthetic polymers can provide this benefit, especially polymers that contain hydrophilic groups, e.g., carboxylate groups. Other polymers that can provide shear-thinning and hydrophilicity include cationic materials that also contain hydrophilic groups and polymers that contain multiple ether linkages. Cationic materials include cationic sugar and/or starch derivatives.

Preferred polymers are those having higher molecular weights, although molecular weights down to about 5,000 can provide some results. In general, the polymers should have molecular weights of more than about 10,000, preferably more than about 100,000, more preferably more than about 250,000, and even more preferably more than about 500,000. The molecular weight should normally be, from about 10,000 to about 100,000; preferably from about 100,000 to about 1,000,000; more preferably from about 1,000,000 to about 4,000,000; and even more preferably greater than 4,000,000 million.

Examples of suitable materials for use herein include polymers preferably selected from the group consisting of xanthan gums, guar gums, gum arabic, pectin poly(styrene sulfonate), and mixtures thereof of monomers and/or polymers. These polymers can also be used in combination with polymers that do not provide the benefit or provide the benefit to lesser extent to achieve an improved end result cleaning. The most preferred is xanthan gum.

The polymer used is preferably one that provides shear-thinning, especially for ease of dispensing. Compositions which are inherently shear-thinning can be used full strength without modification. Hard surface detergent compositions and especially the preferred detergent compositions described herein should have a viscosity of less than about 250 cps, preferably less than about 100 cps, and even more preferably less than about 15. The viscosity is determined using a Brookfield Synchroelectric Viscometer, model LVT, made by Brookfield Engineering Laboratory, Inc., Stoughton,

Massachusetts, using a No. 1 spindle at 60 rpm, and at a temperature of about 20°C. (Constant shear rate of about 13 inverse seconds.)

Shear-thinning characteristics of, e.g., polymers and/or compositions, are determined using a Carrimed Controlled Stress Rheometer Model CSL 100, made by Carrimed Ltd., Interpret House, Curtis Road Estate, Dorking, Surrey RH 4 1DP, England. The Rheometer employs double concentric cylinders geometry to make steady shear measurements at various shear rates. These measurements are made at about 26°C. The shear-thinning, pseudoplastic behavior of the xanthan gum system can be mathematically modeled by the equation:

$$N = KR^{n-1}$$

where N is the apparent viscosity, K is the consistency constant, R is the shear rate, and n is the shear index. For best spraying results (dispensing) the values of K and n should give viscosities below 15 cps at spraying shear rates (~10,000 inversed seconds, as reported in trade literature).

Shear-thinning behavior is described in U.S. Pat. No. 4,783,283, Stoddart, issued Nov. 8, 1988, especially the portion appearing at column 2, line 46, et seq.

The Detergent Surfactant

Detergent surfactants that are used in hard surface cleaner compositions include anionic, nonionic, amphoteric (including zwitterionic), and cationic detergent surfactants and mixtures thereof. Suitable detergents are well known in the art and include those described in U.S. Pat. Nos.: 4,111,854, Spadini et al., issued Sept. 5, 1978; 4,424,408, Imamura et al., issued Jan. 27, 1981; 4,414,128, Goffinet, issued Nov. 8, 1983; 4,612,135, Wenzel, issued Sept. 16, 1986; 4,743,395, Leifheit, issued May 10, 1988; 4,749,509, Kacher, issued June 7, 1988; 4,759,867, Choy et al., issued July 26, 1988; 4,769,172, Siklosi, issued Sept. 6, 1988; 4,804,491, Choy et al., issued Feb. 14, 1989; and 4,895,669, Choy et al., issued Jan. 23, 1990, all of said patents being incorporated herein by reference.

Detergent compositions, or solutions, especially those which are to be used with an implement containing a superabsorbent material, preferred, require sufficient detergent to enable the solution to provide cleaning without overloading the superabsorbent material with solution, but the solutions cannot normally have more than about 0.5% by weight of the solution of detergent surfactant without the performance suffering. Therefore, the level of detergent surfactant in the cleaning solution should be from about 0.01% to about 0.5 %, more preferably from about 0.05% to about 0.4%, and even more preferably from about 0.05% to about 0.3%, by weight of the solution/composition. The preferred solution can also contain one or more solvents to

aid cleaning at a level preferably from about 0.1% to about 5.0%, more preferably from about 0.25% to about 3.0%, and even more preferably from about 0.5% to about 2%, of the solution.

As discussed before, the pH should be more than about 9.3, preferably more than about 10, more preferably more than about 10.3, to aid in cleaning when using conventional systems such as sponges, cloths, and mops such as sponge mops, strip mops, string mops mops, floor cloths, etc., and to avoid hindering absorption when using with pad containg superabsorbent materials, and the alkalinity should preferably be provided, at least in part, by volatile materials, to avoid streaking/filming problems.

The detergent surfactant is preferably linear, e.g., branching and aromatic groups should not be present, and the detergent surfactant is preferably relatively water soluble, e.g., having a hydrophobic chain containing from about 8 to about 14, preferably from about 8 to about 12, carbon atoms, and, for nonionic detergent surfactants, having an HLB of from about 9 to about 14, preferably from about 10 to about 13, more preferably from about 10 to about 12.

The invention also comprises a detergent composition as disclosed herein in a container in association with instructions to use it with an implement comprising an effective amount of a superabsorbent material, and, optionally, in a container in a kit comprising the implement, or, at least, a disposable cleaning pad comprising a superabsorbent material. The invention also relates to the use of the composition and a cleaning pad comprising a suberabsorbent material to effect cleaning of soiled surfaces.

The detergent composition, (cleaning solution) is an aqueous-based solution comprising one or more detergent surfactants, alkaline materials to provide the desired alkaline pH, and optional solvents, builders, chelants, suds suppressors, enzymes, etc. Suitable surfactants include anionic, nonionic, zwitterionic, and amphoteric surfactants as discussed above, preferably anionic and nonionic detergent surfactants having hydrophobic chains containing from about 8 to about 14, preferably from about 8 to about 12, carbon atoms. Examples of anionic surfactants include, but are not limited to, linear alkyl sulfates, alkyl sulfonates, and the like. Examples of nonionic surfactants include alkylethoxylates and the like. Examples of zwitterionic surfactants include betaines and sulfobetaines. Examples of amphoteric surfactants include alkylampho glycines, and alkyl imino propionate. All of the above materials are available commercially, and are described in McCutcheon's Vol. 1: Emulsifiers and Detergents, North American Ed., McCutcheon Division, MC Publishing Co., 1997, incorporated herein by reference.

The Solvent

Suitable solvents include short chain (e.g., C₁-C₆) derivatives of oxyethylene glycol and oxypropylene glycol, such as mono- and di-ethylene glycol n-hexyl ether; mono-, di- and tri-propylene glycol n-butyl ether; and the like. Other volatile solvents such as ethanol, isopropanol and the like are also preferred in the context of the present invention.

The Sud Suppressor

Suitable suds suppressors include silicone polymers and linear or branched C₁₀-C₁₈ fatty acids, paraffins or alcohols. Dow Corning AF (contains: Polyethylene glycol stearate (4% Wt, CAS # 9004993); Methylated silica (2% Wt, CAS # 67762907); Octamethyl cyclotetrasiloxane (2% Wt, CAS # 556672) is preferred..

The suds suppressor at an effective level, typically from about 0.0005 to about 0.02, preferably from about 0.001 to about 0.01, more preferably from about 0.002 to about 0.003%, by weight of the solution/composition, provides a technical improvement in spotting and filming, particularly on ceramic surfaces. The reason for this is the grout lines on ceramic create low spots as the mop moves across, generating suds. If too high a level of suds is generated, it can dry down into streaks. Furthermore, consumer research shows that suds seen on floor during mopping is perceived by some consumers as leading to film/streaking.

Lowering suds on floor during mopping can provide varying degrees of technical and perceptual benefits for not leaving film/streaks. The degree of benefit depends on the level of suds created and to what degree the level of suds is controlled, particularly during mopping.

Known suds suppressors can be used, but it is highly desirable to use a silicone suds suppressor since they are effective at very low levels and therefore can minimize the total water insoluble material needed while having at least an effective amount of suds suppressor present.

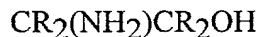
Builders

Suitable builders include those soluble, especially alkali metal, e.g., sodium and/or potassium and/or amine and/or substituted amine, salts of conventional builders, including those derived from phosphorous sources, such as orthophosphate and pyrophosphate, and non-phosphorous sources, such as nitrilotriacetic acid, S,S-ethylene diamine disuccinic acid, and the like. Suitable chelants include ethylenediaminetetraacetic acid and citric acid, and the like.

Optional Ingredients

Suitable enzymes include lipases, proteases, amylases and other enzymes known to be useful for catalysis of soil degradation. The total level of such ingredients is low,

preferably less than about 0.1%, more preferably less than about 0.05%, to avoid causing filming/streaking problems. Preferably, the compositions should be essentially free of materials that cause filming/streaking problems. Accordingly, it is desirable to use alkaline materials that do not cause filming and/or streaking for the majority of the buffering. Suitable alkaline buffers are carbonate, bicarbonate, citrate, etc. The preferred alkaline buffers are alkanol amines having the formula:



wherein each R is selected from the group consisting of hydrogen and alkyl groups containing from one to four carbon atoms and the total of carbon atoms in the compound is from three to six, preferably, 2-amino-2-methylpropanol.

A suitable cleaning solution for use with the present implement comprises from about 0.05% to about 0.3% of detergent surfactant, preferably comprising a linear alcohol ethoxylate detergent surfactant (e.g., Neodol 1-5®, available from Shell Chemical Co.) and an alkylsulfonate (e.g., Bioterge ® PAS-8s, a linear C₈ sulfonate available from Stepan Co.); from about 0.5 to about 2.0% propylene glycol n-butyl ether (Dow Co.), from about 0.5% to about 3.0% ethanol (Quantum chemicals), from about 0.05% to about 0.25%, of volatile alkaline material, e.g., 2-amino-2-methyl-1-propanol; optional adjuvants such as dyes and/or perfumes; and from about 99.9% to about 90% deionized or softened water.

II. The Implement Plus Cleaning Pad System

The implement plus cleaning pad system in the preferred embodiment is based on providing convenience. Therefore, it is preferable to use an implement which comprises a cleaning pad, preferably removable and/or disposable, that contains a superabsorbent material and which preferably also provides significant cleaning benefits. The preferred cleaning performance benefits are related to the preferred structural characteristics described below, combined with the ability of the pad to work synergistically with the present invention to remove soils. The cleaning pad, as described herein requires the use of the detergent composition, as described hereinafter, to provide optimum performance.

The cleaning pads will preferably have an absorbent capacity when measured under a confining pressure of 0.09 psi after 20 minutes (1200 seconds) (hereafter referred to as "t₁₂₀₀ absorbent capacity") of at least about 10 g deionized water per g of the cleaning pad. The absorbent capacity of the pad is measured at 20 minutes (1200 seconds) after exposure to deionized water, as this represents a typical time for the consumer to clean a hard surface such as a floor. The confining pressure represents typical pressures exerted on the pad during the cleaning process. As such, the cleaning

pad should be capable of absorbing significant amounts of the cleaning solution within this 1200 second period under 0.09 psi. The cleaning pad will preferably have a t_{1200} absorbent capacity of at least about 15 g/g, more preferably at least about 20 g/g, still more preferably at least about 25 g/g and most preferably at least about 30 g/g. The cleaning pad will preferably have a t_{900} absorbent capacity of at least about 10 g/g, more preferably a t_{900} absorbent capacity of at least about 20 g/g.

Values for t_{1200} and t_{900} absorbent capacity are measured by the performance under pressure (referred to herein as "PUP") method, which is described in detail in the Test Methods section below.

The cleaning pads will also preferably, but not necessarily, have a total fluid capacity (of deionized water) of at least about 100 g, more preferably at least about 200 g, still more preferably at least about 300 g and most preferably at least about 400 g. While pads having a total fluid capacity less than 100 g are within the scope of the invention, they are not as well suited for cleaning large areas, such as seen in a typical household, as are higher capacity pads.

Absorbent Layer

The absorbent layer serves to retain any fluid and soil absorbed by the cleaning pad during use. While the preferred scrubbing layer, described hereinafter, has some affect on the pad's ability to absorb fluid, the absorbent layer plays the major role in achieving the desired overall absorbency. Furthermore, the absorbent layer preferably comprises multiple layers which are designed to provide the cleaning pad with multiple planar surfaces.

From a fluid absorbency perspective, the absorbent layer will be capable of removing fluid and soil from any "scrubbing layer" so that the scrubbing layer will have capacity to continually remove soil from the surface. The absorbent layer also should be capable of retaining absorbed material under typical in-use pressures to avoid "squeeze-out" of absorbed soil, cleaning solution, etc.

The absorbent layer can comprise any material that is capable of absorbing and retaining fluid during use. To achieve desired total fluid capacities, it is preferred to include in the absorbent layer a material having a relatively high capacity (in terms of grams of fluid per gram of absorbent material). As used herein, the term "superabsorbent material" means any absorbent material having a g/g capacity for water of at least about 15 g/g, when measured under a confining pressure of 0.3 psi. The cleaning solutions (compositions) disclosed above are aqueous based, so it is preferred that the superabsorbent materials have a relatively high g/g capacity for water or water-based fluids.

Representative superabsorbent materials include water insoluble, water-swellaable superabsorbent gelling polymers (referred to herein as "superabsorbent gelling polymers") which are well known in the literature. These materials demonstrate very high absorbent capacities for water. The superabsorbent gelling polymers useful in the present invention can have a size, shape and/or morphology varying over a wide range. These polymers can be in the form of particles that do not have a large ratio of greatest dimension to smallest dimension (e.g., granules, flakes, pulverulents, interparticle aggregates, interparticle crosslinked aggregates, and the like) or they can be in the form of fibers, sheets, films, foams, laminates, and the like. The use of superabsorbent gelling polymers in fibrous form provides the benefit of providing enhanced retention of the superabsorbent material, relative to particles, during the cleaning process. While their capacity is generally lower for aqueous-based mixtures, these materials still demonstrate significant absorbent capacity for such mixtures. The patent literature is replete with disclosures of water-swellaable materials. See, for example, U.S. Patent 3,699,103 (Harper et al.), issued June 13, 1972; U.S. Patent 3,770,731 (Harmon), issued June 20, 1972; U.S. Reissue Patent 32,649 (Brandt et al.), reissued April 19, 1989; U.S. Patent 4,834,735 (Alemany et al.), issued May 30, 1989, said patents being incorporated herein by reference.

Superabsorbent gelling polymers useful in the present invention include a variety of water-insoluble, but water-swellaable polymers capable of absorbing large quantities of fluids. Such polymeric materials are also commonly referred to as "hydrocolloids", and can include polysaccharides such as carboxymethyl starch, carboxymethyl cellulose, and hydroxypropyl cellulose; nonionic types such as polyvinyl alcohol, and polyvinyl ethers; cationic types such as polyvinyl pyridine, polyvinyl morpholinone, and N,N-dimethylaminoethyl or N,N-diethylaminopropyl acrylates and methacrylates, and the respective quaternary salts thereof. Typically, superabsorbent gelling polymers that are useful have a multiplicity of anionic functional groups, such as sulfonic acid, and more typically carboxy, groups. Examples of polymers suitable for use herein include those which are prepared from polymerizable, unsaturated, acid-containing monomers. Thus, such monomers include the olefinically unsaturated acids and anhydrides that contain at least one carbon to carbon olefinic double bond. More specifically, these monomers can be selected from olefinically unsaturated carboxylic acids and acid anhydrides, olefinically unsaturated sulfonic acids, and mixtures thereof.

Some non-acid monomers can also be included, usually in minor amounts, in preparing the superabsorbent gelling polymers useful herein. Such non-acid monomers can include, for example, the water-soluble or water-dispersible esters of the acid-

containing monomers, as well as monomers that contain no carboxylic or sulfonic acid groups at all. Optional non-acid monomers can thus include monomers containing the following types of functional groups: carboxylic acid or sulfonic acid esters, hydroxyl groups, amide-groups, amino groups, nitrile groups, quaternary ammonium salt groups, aryl groups (e.g., phenyl groups, such as those derived from styrene monomer). These non-acid monomers are well-known materials and are described in greater detail, for example, in U.S. Patent 4,076,663 (Masuda et al), issued February 28, 1978, and in U.S. Patent 4,062,817 (Westerman), issued December 13, 1977, both of which are incorporated by reference.

Olefinically unsaturated carboxylic acid and carboxylic acid anhydride monomers include the acrylic acids typified by acrylic acid itself, methacrylic acid, ethacrylic acid, α -chloroacrylic acid, α -cyanoacrylic acid, β -methylacrylic acid (crotonic acid), α -phenylacrylic acid, β -acryloxypropionic acid, sorbic acid, α -chlorosorbic acid, angelic acid, cinnamic acid, p-chlorocinnamic acid, β -styrylacrylic acid, itaconic acid, citraconic acid, mesaconic acid, glutaconic acid, aconitic acid, maleic acid, fumaric acid, tricarboxyethylene and maleic acid anhydride.

Olefinically unsaturated sulfonic acid monomers include aliphatic or aromatic vinyl sulfonic acids such as vinylsulfonic acid, allyl sulfonic acid, vinyl toluene sulfonic acid and styrene sulfonic acid; acrylic and methacrylic sulfonic acid such as sulfoethyl acrylate, sulfoethyl methacrylate, sulfopropyl acrylate, sulfopropyl methacrylate, 2-hydroxy-3-methacryloxypropyl sulfonic acid and 2-acrylamide-2-methylpropane sulfonic acid.

Preferred superabsorbent gelling polymers for use in the present invention contain carboxy groups. These polymers include hydrolyzed starch-acrylonitrile graft copolymers, partially neutralized hydrolyzed starch-acrylonitrile graft copolymers, starch-acrylic acid graft copolymers, partially neutralized starch-acrylic acid graft copolymers, saponified vinyl acetate-acrylic ester copolymers, hydrolyzed acrylonitrile or acrylamide copolymers, slightly network crosslinked polymers of any of the foregoing copolymers, partially neutralized polyacrylic acid, and slightly network crosslinked polymers of partially neutralized polyacrylic acid. These polymers can be used either solely or in the form of a mixture of two or more different polymers. Examples of these polymer materials are disclosed in U.S. Patent 3,661,875, U.S. Patent 4,076,663, U.S. Patent 4,093,776, U.S. Patent 4,666,983, and U.S. Patent 4,734,478, all of said patents being incorporated herein by reference.

Most preferred polymer materials for use in making the superabsorbent gelling polymers are slightly networked crosslinked polymers of partially neutralized polyacrylic

acids and starch derivatives thereof. Most preferably, the hydrogel-forming absorbent polymers comprise from about 50% to about 95%, preferably about 75%, neutralized, slightly network crosslinked, polyacrylic acid (i.e. poly (sodium acrylate/acrylic acid)). Network crosslinking renders the polymer substantially water-insoluble and, in part, determines the absorptive capacity and extractable polymer content characteristics of the superabsorbent gelling polymers. Processes for network crosslinking these polymers and typical network crosslinking agents are described in greater detail in U.S. Patent 4,076,663, incorporated herein by reference.

While the superabsorbent gelling polymers are preferably of one type (i.e., homogeneous), mixtures of polymers can also be used in the implements used in the preferred embodiment. For example, mixtures of starch-acrylic acid graft copolymers and slightly network crosslinked polymers of partially neutralized polyacrylic acid can be used in the present invention.

While any of the superabsorbent gelling polymers described in the prior art can be useful in the present invention, it has recently been recognized that where significant levels (e.g., more than about 50% by weight of the absorbent structure) of superabsorbent gelling polymers are to be included in an absorbent structure, and in particular where one or more regions of the absorbent layer will comprise more than about 50%, by weight of the region, the problem of gel blocking by the swollen particles may impede fluid flow and thereby adversely affect the ability of the gelling polymers to absorb to their full capacity in the desired period of time. U.S. Patent 5,147,343 (Kellenberger et al.), issued September 15, 1992 and U.S. Patent 5,149,335 (Kellenberger et al.), issued September 22, 1992, said patents being incorporated herein by reference, describe superabsorbent gelling polymers in terms of their Absorbency Under Load (AUL), where gelling polymers absorb fluid (0.9% saline) under a confining pressure of 0.3 psi. (The disclosure of each of these patents is incorporated herein.) The methods for determining AUL are described in these patents. Polymers described therein can be particularly useful in embodiments of the present invention that contain regions of relatively high levels of superabsorbent gelling polymers. In particular, where high concentrations of superabsorbent gelling polymer are incorporated in the cleaning pad, those polymers will preferably have an AUL, measured according to the methods described in U.S. Patent 5,147,343, incorporated herein by reference, of at least about 24 ml/g, more preferably at least about 27 ml/g after 1 hour; or an AUL, measured according to the methods described in U.S. Patent 5,149,335, incorporated herein by reference, of at least about 15 ml/g, more preferably at least about 18 ml/g after 15 minutes. Commonly assigned copending U.S. application Serial Numbers 08/219,547 (Goldman et al.), filed March 29,

1994 and 08/416,396 (Goldman et al.), filed April 6, 1995 (both of which are incorporated by reference herein), also address the problem of gel blocking and describe superabsorbent gelling polymers useful in overcoming this phenomena. These applications specifically describe superabsorbent gelling polymers which avoid gel blocking at even higher confining pressures, specifically 0.7 psi. In the embodiments of the present invention where the absorbent layer will contain regions comprising high levels (e.g., more than about 50% by weight of the region) of superabsorbent gelling polymer, it may be preferred that the superabsorbent gelling polymer be as described in the aforementioned applications by Goldman et al.

Other useful superabsorbent materials include hydrophilic polymeric foams, such as those described in commonly assigned copending U.S. patent application Serial No. 08/563,866 (DesMarais et al.), filed November 29, 1995 and U.S. Patent No. 5,387,207 (Dyer et al.), issued February 7, 1995, said patents being incorporated herein by reference. These references describe polymeric, hydrophilic absorbent foams that are obtained by polymerizing a high internal phase water-in-oil emulsion (commonly referred to as HIPEs). These foams are readily tailored to provide varying physical properties (pore size, capillary suction, density, etc.) that affect fluid handling ability. As such, these materials are particularly useful, either alone or in combination with other such foams or with fibrous structures, in providing the overall capacity.

Where superabsorbent material is included in the absorbent layer, the absorbent layer will preferably comprise at least about 15%, by weight of the absorbent layer, more preferably at least about 20%, still more preferably at least about 25%, of the superabsorbent material.

The absorbent layer can also consist of or comprise fibrous material. Fibers useful in the present invention include those that are naturally occurring (modified or unmodified), as well as synthetically made fibers. Examples of suitable unmodified/modified naturally occurring fibers include cotton, Esparto grass, bagasse, hemp, flax, silk, wool, wood pulp, chemically modified wood pulp, jute, ethyl cellulose, and cellulose acetate. Suitable synthetic fibers can be made from polyvinyl chloride, polyvinyl fluoride, polytetrafluoroethylene, polyvinylidene chloride, polyacrylics such as ORLON®, polyvinyl acetate, RAYON®, polyethylvinyl acetate, non-soluble or soluble polyvinyl alcohol, polyolefins such as polyethylene (e.g., PULPEX®) and polypropylene, polyamides such as nylon, polyesters such as DACRON® or KODEL®, polyurethanes, polystyrenes, and the like. The absorbent layer can comprise solely naturally occurring fibers, solely synthetic fibers, or any compatible combination of naturally occurring and synthetic fibers.

The fibers useful herein can be hydrophilic, hydrophobic or can be a combination of both hydrophilic and hydrophobic fibers. As indicated above, the particular selection of hydrophilic or hydrophobic fibers will depend upon the other materials included in the absorbent (and to some degree the scrubbing) layer. That is, the nature of the fibers are preferably such that the cleaning pad exhibits the preferred fluid delay and overall fluid absorbency. Suitable hydrophilic fibers for use in the present invention include cellulosic fibers, modified cellulosic fibers, rayon, polyester fibers such as hydrophilic nylon (HYDROFIL®). Suitable hydrophilic fibers can also be obtained by hydrophilizing hydrophobic fibers, such as surfactant-treated or silica-treated thermoplastic fibers derived from, for example, polyolefins such as polyethylene or polypropylene, polyacrylics, polyamides, polystyrenes, polyurethanes and the like.

Suitable wood pulp fibers can be obtained from well-known chemical processes such as the Kraft and sulfite processes. It is especially preferred to derive these wood pulp fibers from southern soft woods due to their premium absorbency characteristics. These wood pulp fibers can also be obtained from mechanical processes, such as ground wood, refiner mechanical, thermomechanical, chemimechanical, and chemi-thermomechanical pulp processes. Recycled or secondary wood pulp fibers, as well as bleached and unbleached wood pulp fibers, can be used.

Another type of hydrophilic fiber for use in the present invention is chemically stiffened cellulosic fibers. As used herein, the term "chemically stiffened cellulosic fibers" means cellulosic fibers that have been stiffened by chemical means to increase the stiffness of the fibers under both dry and aqueous conditions. Such means can include the addition of a chemical stiffening agent that, for example, coats and/or impregnates of the fibers. Such means can also include the stiffening of the fibers by altering the chemical structure, e.g., by crosslinking polymer chains.

Where fibers are used as the absorbent layer (or a constituent component thereof), the fibers can optionally be combined with a thermoplastic material. Upon melting, at least a portion of this thermoplastic material migrates to the intersections of the fibers, typically due to interfiber capillary gradients. These intersections become bond sites for the thermoplastic material. When cooled, the thermoplastic materials at these intersections solidify to form the bond sites that hold the matrix or web of fibers together in each of the respective layers. This can be beneficial in providing additional overall integrity to the cleaning pad.

Amongst its various effects, bonding at the fiber intersections increases the overall compressive modulus and strength of the resulting thermally bonded member. In the case of the chemically stiffened cellulosic fibers, the melting and migration of the

thermoplastic material also has the effect of increasing the average pore size of the resultant web, while maintaining the density and basis weight of the web as originally formed. This can improve the fluid acquisition properties of the thermally bonded web upon initial exposure to fluid, due to improved fluid permeability, and upon subsequent exposure, due to the combined ability of the stiffened fibers to retain their stiffness upon wetting and the ability of the thermoplastic material to remain bonded at the fiber intersections upon wetting and upon wet compression. In net, thermally bonded webs of stiffened fibers retain their original overall volume, but with the volumetric regions previously occupied by the thermoplastic material becoming open to thus increase the average interfiber capillary pore size.

Thermoplastic materials useful in the present invention can be in any of a variety of forms including particulates, fibers, or combinations of particulates and fibers. Thermoplastic fibers are a particularly preferred form because of their ability to form numerous interfiber bond sites. Suitable thermoplastic materials can be made from any thermoplastic polymer that can be melted at temperatures that will not extensively damage the fibers that comprise the primary web or matrix of each layer. Preferably, the melting point of this thermoplastic material will be less than about 190°C, and preferably between about 75°C and about 175°C. In any event, the melting point of this thermoplastic material should be no lower than the temperature at which the thermally bonded absorbent structures, when used in the cleaning pads, are likely to be stored. The melting point of the thermoplastic material is typically no lower than about 50°C.

The thermoplastic materials, and in particular the thermoplastic fibers, can be made from a variety of thermoplastic polymers, including polyolefins such as polyethylene (e.g., PULPEX®) and polypropylene, polyesters, copolyesters, polyvinyl acetate, polyethylvinyl acetate, polyvinyl chloride, polyvinylidene chloride, polyacrylics, polyamides, copolyamides, polystyrenes, polyurethanes and copolymers of any of the foregoing such as vinyl chloride/vinyl acetate, and the like. Depending upon the desired characteristics for the resulting thermally bonded absorbent member, suitable thermoplastic materials include hydrophobic fibers that have been made hydrophilic, such as surfactant-treated or silica-treated thermoplastic fibers derived from, for example, polyolefins such as polyethylene or polypropylene, polyacrylics, polyamides, polystyrenes, polyurethanes and the like. The surface of the hydrophobic thermoplastic fiber can be rendered hydrophilic by treatment with surfactant, such as a nonionic and/or anionic surfactant, e.g., by spraying the fiber with surfactant, by dipping the fiber into a surfactant or by including the surfactant as part of the polymer melt in producing the thermoplastic fiber. Upon melting and resolidification, the surfactant will tend to remain

at the surfaces of the thermoplastic fiber. Suitable surfactants include nonionic surfactants such as Brij[®] 76 manufactured by ICI Americas, Inc. of Wilmington, Delaware, and various surfactants sold under the Pegosperse[®] trademark by Glyco Chemical, Inc. of Greenwich, Connecticut. Besides nonionic surfactants, anionic surfactants can also be used. These surfactants can be applied to the thermoplastic fibers at levels of, for example, from about 0.2 to about 1 g. per sq. of centimeter of thermoplastic fiber.

Suitable thermoplastic fibers can be made from a single polymer (monocomponent fibers), or can be made from more than one polymer (e.g., bicomponent fibers). As used herein, "bicomponent fibers" refers to thermoplastic fibers that comprise a core fiber made from one polymer that is encased within a thermoplastic sheath made from a different polymer. The polymer comprising the sheath often melts at a different, typically lower, temperature than the polymer comprising the core. As a result, these bicomponent fibers provide thermal bonding due to melting of the sheath polymer, while retaining the desirable strength characteristics of the core polymer.

Suitable bicomponent fibers for use herein can include sheath/core fibers having the following polymer combinations: polyethylene/polypropylene, polyethylvinyl acetate/polypropylene, polyethylene/polyester, polypropylene/polyester, copolyester/polyester, and the like. Particularly suitable bicomponent thermoplastic fibers for use herein are those having a polypropylene or polyester core, and a lower melting copolyester, polyethylvinyl acetate or polyethylene sheath (e.g., those available from Danaklon a/s, Chisso Corp., and CELBOND[®], available from Hercules). These bicomponent fibers can be concentric or eccentric. As used herein, the terms "concentric" and "eccentric" refer to whether the sheath has a thickness that is even, or uneven, through the cross-sectional area of the bicomponent fiber. Eccentric bicomponent fibers can be desirable in providing more compressive strength at lower fiber thicknesses.

Methods for preparing thermally bonded fibrous materials are described in co-pending U.S. application Serial No. 08/479,096 (Richards et al.), filed July 3, 1995 (see especially pages 16-20) and U.S. Patent 5,549,589 (Horney et al.), issued August 27, 1996 (see especially Columns 9 to 10). The disclosure of both of these references are incorporated by reference herein.

The absorbent layer can also comprise a HIPE-derived hydrophilic, polymeric foam that does not have the high absorbency of those described above as "superabsorbent materials". Such foams and methods for their preparation are described in U.S. Patent 5,550,167 (DesMarais), issued August 27, 1996; and commonly assigned copending

U.S. patent application Serial No. 08/370,695 (Stone et al.), filed January 10, 1995 (both of which are incorporated by reference herein).

The absorbent layer of the cleaning pad can be comprised of a homogeneous material, such as a blend of cellulosic fibers (optionably thermally bonded) and swellable superabsorbent gelling polymer. Alternatively, the absorbent layer can be comprised of discrete layers of material, such as a layer of thermally bonded airlaid material and a discrete layer of a superabsorbent material. For example, a thermally bonded layer of cellulosic fibers can be located lower than (i.e., beneath) the superabsorbent material (i.e., between the superabsorbent material and the scrubbing layer). In order to achieve high absorptive capacity and retention of fluids under pressure, while at the same time providing initial delay in fluid uptake, it may be preferable to utilize such discrete layers when forming the absorbent layer. In this regard, the superabsorbent material can be located remote from the scrubbing layer by including a less absorbent layer as the lowermost aspect of the absorbent layer. For example, a layer of cellulosic fibers can be located lower (i.e., beneath) than the superabsorbent material (i.e., between the superabsorbent material and the scrubbing layer).

In a preferred embodiment, the absorbent layer will comprise a thermally bonded airlaid web of cellulose fibers (Flint River, available from Weyerhaeuser, Wa) and AL Thermal C (thermoplastic available from Danaklon a/s, Varde, Denmark), and a swellable hydrogel-forming superabsorbent polymer. The superabsorbent polymer is preferably incorporated such that a discrete layer is located near the surface of the absorbent layer which is remote from the scrubbing layer. Preferably, a thin layer of, e.g., cellulose fibers (optionally thermally bonded) are positioned above the superabsorbent gelling polymer to enhance containment.

B. Optional, but Preferred, Scrubbing Layer

The scrubbing layer is the portion of the cleaning pad that contacts the soiled surface during cleaning. As such, materials useful as the scrubbing layer are preferably sufficiently durable that the layer will retain its integrity during the cleaning process. In addition, when the cleaning pad is used in combination with a solution, the scrubbing layer is preferably capable of absorbing liquids and soils, and relinquishing those liquids and soils to the absorbent layer. This will ensure that the scrubbing layer will continually be able to remove additional material from the surface being cleaned. Whether the implement is used with a cleaning solution (i.e., in the wet state) or without cleaning solution (i.e., in the dry state), the scrubbing layer will, in addition to removing particulate matter, facilitate other functions, such as polishing, dusting, and buffing the surface being cleaned.

The scrubbing layer can be a monolayer, or a multi-layer structure one or more of whose layers can be slitted to facilitate the scrubbing of the soiled surface and the uptake of particulate matter. This scrubbing layer, as it passes over the soiled surface, interacts with the soil (and cleaning solution when used), loosening and emulsifying tough soils and permitting them to pass freely into the absorbent layer of the pad. The scrubbing layer preferably contains openings (e.g., slits) that provide an easy avenue for larger particulate soil to move freely in and become entrapped within the absorbent layer of the pad. Low density structures are preferred for use as the scrubbing layer, to facilitate transport of particulate matter to the pad's absorbent layer.

In order to provide desired integrity, materials particularly suitable for the scrubbing layer include synthetics such as polyolefins (e.g., polyethylene and polypropylene), polyesters, polyamides, synthetic cellulose (e.g., RAYON®), and blends thereof. Such synthetic materials can be manufactured using a known process such as carded, spunbond, meltblown, airlaid, needlepunched and the like.

C. Optional Attachment Layer

The cleaning pads of the present invention can optionally have an attachment layer that allows the pad to be connected to an implement's handle or the support head in preferred implements. The attachment layer will be necessary in those embodiments where the absorbent layer is not suitable for attaching the pad to the support head of the handle. The attachment layer can also function as a means to prevent fluid flow through the top surface (i.e., the handle-contacting surface) of the cleaning pad, and can further provide enhanced integrity of the pad. As with the scrubbing and absorbent layers, the attachment layer can consist of a mono-layer or a multi-layer structure, so long as it meets the above requirements.

In a preferred embodiment of the present invention, the attachment layer will comprise a surface which is capable of being mechanically attached to the handle's support head by use of known hook and loop technology. In such an embodiment, the attachment layer will comprise at least one surface which is mechanically attachable to hooks that are permanently affixed to the bottom surface of the handle's support head.

To achieve the desired fluid imperviousness and attachability, it is preferred that a laminated structure comprising, e.g., a meltblown film and fibrous, nonwoven structure be utilized. In a preferred embodiment, the attachment layer is a tri-layered material having a layer of meltblown polypropylene film located between two layers of spunbonded polypropylene.

D. Optional, but Preferred, Multiple Planar Surfaces

While the ability of the cleaning pad to absorb and retain fluids has been determined to be important to hard surface cleaning performance (see, e.g., copending U.S. Patent Application Serial No. 08/756,507 (Holt et al.), copending U.S. Patent Application Serial No. 08/756,864 (Sherry et al.), and copending U.S. Patent Application Serial No. 08/756,999 (Holt et al.), all filed November 26, 1996 and incorporated by reference herein.), preferred performance can be achieved by properly defining the overall structure of the cleaning pad. In particular, pads having an essentially flat floor contacting surface (i.e., essentially one planar surface for contacting the soiled surface during cleaning) do not provide the best performance because soil tends to build up on the leading edge, which also is the main point where the cleaning solution is transferred to the absorbent layer.

The preferred pads provide multiple planar surfaces during cleaning and provide enhanced performance. Referring to Figure 2 in the drawings, cleaning pad 100 is depicted as having an upper surface 103 that allows the pad to be releasably attached to a handle. Cleaning pad 100 also has a lower surface depicted generally as 110 which contacts the floor or other hard surface during cleaning. This lower surface 110 actually consists of 3 substantially planar surfaces 112, 114 and 116. As depicted, the planes corresponding to surfaces 112 and 116 intersect the plane corresponding to surface 114. Thus, when an implement to which pad 100 is attached is moved from rest in the direction indicated by Y_f , friction causes pad 100 to "rock" such that lower surface 112 contacts the surface being cleaned. As the movement in the Y_f direction diminishes, lower surface 114 will then contact the surface being cleaned. As the implement and pad are moved from rest in the Y_b direction, friction causes pad 100 to rock such that lower surface 116 then contacts the surface being cleaned. As this cleaning motion is repeated, the portion of the pad contacting the soiled surface are constantly changing.

It is believed that the enhanced cleaning of the preferred pads is in-part due to the "lifting" action that results from the back and forth motion during cleaning. In particular, when the cleaning motion in one direction is stopped and the forces exerted on the implement allow pad 100 to "rock" such that the surface-contacting planar surface moves from surface 112 (or 116) to surface 114, soil is moved in an upward direction.

The cleaning pad of the present invention should be capable of retaining absorbed fluid, even during the pressures exerted during the cleaning process. This is referred to herein as the cleaning pad's ability to avoid "squeeze-out" of absorbed fluid, or conversely its ability to retain absorbed fluid under pressure. The method for measuring squeeze-out is described in the Test Methods section. Briefly, the test measures the ability of a saturated cleaning pad to retain fluid when subjected to a pressure of 0.25 psi.

Preferably, the cleaning pads of the present invention will have a squeeze-out value of not more than about 40%, more preferably not more than about 25%, still more preferably not more than about 15%, and most preferably not more than about 10%.

III. Cleaning Implements

The detergent compositions described above can be desirably used with an implement for cleaning a surface, the implement comprising:

- a. a handle; and
- b. a removable cleaning pad containing an effective amount of a superabsorbent material, and having a plurality of substantially planar surfaces, wherein each of the substantially planar surfaces contacts the surface being cleaned, more preferably said pad is a removable cleaning pad having a length and a width, the pad comprising
 - i. a scrubbing layer; and
 - ii. an absorbent layer comprising a first layer and a second layer, where the first layer is located between the scrubbing layer and the second layer (i.e., the first layer is below the second layer) and has a smaller width than the second layer.

An important aspect of the cleaning performance provided by the preferred pad is related to the ability to provide multiple planar surfaces that contact the soiled surface during the cleaning operation. In the context of a cleaning implement such as a mop, these planar surfaces are provided such that during the typical cleaning operation (i.e., where the implement is moved back and forth in a direction substantially parallel to the pad's Y-dimension or width), each of the planar surfaces contact the surface being cleaned as a result of "rocking" of the cleaning pad. This aspect of the invention, and the benefits provided, are discussed in detail with reference to the drawings.

The skilled artisan will recognize that various materials can be utilized to carry out the claimed invention. Thus, while preferred materials are described below for the various implement and cleaning pad components, it is recognized that the scope of the invention is not limited to such disclosures.

a. The Handle

The handle of the above cleaning implement can be any material that will facilitate gripping of the cleaning implement. The handle of the cleaning implement will preferably comprise any elongated, durable material that will provide practical cleaning. The length of the handle will be dictated by the end-use of the implement.

The handle will preferably comprise at one end a support head to which the cleaning pad can be releasably attached. To facilitate ease of use, the support head can be pivotably attached to the handle using known joint assemblies. Any suitable means for attaching the cleaning pad to the support head can be utilized, so long as the cleaning

pad remains afixed during the cleaning process. Examples of suitable fastening means include clamps, hooks & loops (e.g., VELCRO®), and the like. In a preferred embodiment, the support head will comprise hooks on its lower surface that will mechanically attach to the upper layer (preferably a distinct attachment layer) of the absorbent cleaning pad.

A preferred handle, comprising a fluid dispensing means, is depicted in Figure 1 and is fully described in co-pending U.S. Patent Application Serial No. 08/756,774, filed November 15, 1996 by V. S. Ping, et al. (Case 6383), which is incorporated by reference herein. Another preferred handle, which does not contain a fluid dispensing means, is depicted in Figs. 1a and 1b, and is fully described in co-pending U.S. Patent Application Ser. No. 08/716,755, filed September 23, 1996 by A. J. Irwin (P&G Case 6262), which is incorporated by reference herein.

b. The Cleaning Pad

The cleaning pads described hereinbefore can be used without attachment to a handle, or as part of the above cleaning implement. They can therefore be constructed without the need to be attachable to a handle, i.e., such that they can be used either in combination with the handle or as a stand-alone product. As such, it can be preferred to prepare the pads with an optional attachment layer as described hereinbefore. With the exception of an attachment layer, the pads themselves are as described above.

As used herein, the term "direct fluid communication" means that fluid can transfer readily between two cleaning pad components or layers (e.g., the scrubbing layer and the absorbent layer) without substantial accumulation, transport, or restriction by an interposed layer. For example, tissues, nonwoven webs, construction adhesives, and the like can be present between the two distinct components while maintaining "direct fluid communication", as long as they do not substantially impede or restrict fluid as it passes from one component or layer to another.

As used herein, the term "Z-dimension" refers to the dimension orthogonal to the length and width of the cleaning pad of the present invention, or a component thereof. The Z-dimension usually corresponds to the thickness of the cleaning pad or a pad component.

As used herein, the term "X-Y dimension" refers to the plane orthogonal to the thickness of the cleaning pad, or a component thereof. The X and Y dimensions usually correspond to the length and width, respectively, of the cleaning pad or a pad component. In general, when the cleaning pad is used in conjunction with a handle, the implement will be moved in a direction parallel to Y-dimension of the pad. (See the discussion below.)

As used herein, the term "layer" refers to a member or component of a cleaning pad whose primary dimension is X-Y, i.e., along its length and width. It should be understood that the term layer is not necessarily limited to single layers or sheets of material. Thus the layer can comprise laminates or combinations of several sheets or webs of the requisite type of materials. Accordingly, the term "layer" includes the terms "layers" and "layered."

As used herein, the term "hydrophilic" is used to refer to surfaces that are wettable by aqueous fluids deposited thereon. Hydrophilicity and wettability are typically defined in terms of contact angle and the surface tension of the fluids and solid surfaces involved. This is discussed in detail in the American Chemical Society publication entitled Contact Angle, Wettability and Adhesion, edited by Robert F. Gould (Copyright 1964), which is hereby incorporated herein by reference. A surface is said to be wetted by a fluid (i.e., hydrophilic) when either the contact angle between the fluid and the surface is less than 90°, or when the fluid tends to spread spontaneously across the surface, both conditions normally co-existing. Conversely, a surface is considered to be "hydrophobic" if the contact angle is greater than 90° and the fluid does not spread spontaneously across the surface.

As used herein, the term "scrim" means any durable material that provides texture to the surface-contacting side of the cleaning pad's scrubbing layer, and also has a sufficient degree of openness to allow the requisite movement of fluid to the absorbent layer of the cleaning pad. Suitable materials include materials that have a continuous, open structure, such as synthetic and wire mesh screens. The open areas of these materials can be readily controlled by varying the number of interconnected strands that comprise the mesh, by controlling the thickness of those interconnected strands, etc. Other suitable materials include those where texture is provided by a discontinuous pattern printed on a substrate. In this aspect, a durable material (e.g., a synthetic) can be printed on a substrate in a continuous or discontinuous pattern, such as individual dots and/or lines, to provide the requisite texture. Similarly, the continuous or discontinuous pattern can be printed onto a release material that will then act as the scrim. These patterns can be repeating or they can be random. It will be understood that one or more of the approaches described for providing the desired texture can be combined to form the optional scrim material. The Z direction height and open area of the scrim and or scrubbing substrate layer help to control and or retard the flow of liquid into the absorbent core material. The Z height of the scrim and or scrubbing substrate help provide a means of controlling the volume of liquid in contact with the cleaning surface

while at the same time controlling the rate of liquid absorption, fluid communication into the absorption core material.

For purposes of the present invention, an "upper" layer of a cleaning pad is a layer that is relatively further away from the surface that is to be cleaned (i.e., in the implement context, relatively closer to the implement handle during use). The term "lower" layer conversely means a layer of a cleaning pad that is relatively closer to the surface that is to be cleaned (i.e., in the implement context, relatively further away from the implement handle during use). As such, the scrubbing layer is the lower-most layer and the absorbent layer is an upper layer relative to the scrubber layer. The terms "upper" and "lower" are similarly used when referring to layers that are multi-ply (e.g., when the scrubbing layer is a two-ply material). The terms "above" and "below" are used to describe relative locations of two or more materials in a cleaning pad's thickness. By way of illustration, a material A is "above" material B if material B is positioned closer to the scrubbing layer than material A. Similarly, material B is "below" material A in this illustration.

All percentages, ratios and proportions used herein are by weight unless otherwise specified and all numerical limits are the normal approximations within normal limits of accuracy.

IV. Other Embodiments of the Cleaning Pad

To enhance the pad's ability to remove tough soil residues and increase the amount of cleaning fluid in contact with the cleaning surface, it can be desirable to incorporate a scrim material into the cleaning pad. The scrim will be comprised of a durable, tough material that will provide texture to the pad's scrubbing layer, particularly when in-use pressures are applied to the pad. Preferably, the scrim will be located such that it is in close proximity to the surface being cleaned. Thus, the scrim can be incorporated as part of the scrubbing layer or the absorbent layer; or it can be included as a distinct layer, preferably positioned between the scrubbing and absorbent layers. In one preferred embodiment, where the scrim material is of the same X-Y dimension as the overall cleaning pad, it is preferred that the scrim material be incorporated such that it does not directly contact, to a significant degree, the surface being cleaned. This will maintain the ability of the pad to move readily across the hard surface and will aid in preventing non-uniform removal of the cleaning solution employed. As such, if the scrim is part of the scrubbing layer, it will be an upper layer of this component. Of course, the scrim should at the same time be positioned sufficiently low in the pad to provide it's scrubbing function. Thus, if the scrim is incorporated as part of the absorbent

layer, it will be a lower layer thereof. In a separate embodiment, it can be desirable to place the scrim such that it will be in direct contact with the surface to be cleaned.

In addition to the importance of properly positioning the scrim is that the scrim not significantly impede fluid flow through the pad. The scrim therefore is a relatively open web.

The scrim material will be any material that can be processed to provide a tough, open-textured web. Such materials include polyolefins (e.g., polyethylene, polypropylene), polyesters, polyamides, and the like. The skilled artisan will recognize that these different materials exhibit a different degree of hardness. Thus, the hardness of the scrim material can be controlled, depending on the end-use of the pad/implement. Where the scrim is incorporated as a discrete layer, many commercial sources of such materials are available (e.g., design number VO1230, available from Conwed Plastics, Minneapolis, MN). Alternatively, the scrim can be incorporated by printing a resin or other synthetic material (e.g. latex) onto a substrate, such as is disclosed in U.S. Patent No. 4,745,021, issued May 17, 1988 to Ping, III et al., and U.S. Patent No. 4,733,774, issued March 29, 1988 to Ping, III et al., both of which are incorporated by reference herein.

The various layers that comprise the cleaning pad can be bonded together utilizing any means that provides the pad with sufficient integrity during the cleaning process. The scrubbing and attachment layers can be bonded to the absorbent layer or to each other by any of a variety of bonding means, including the use of a uniform continuous layer of adhesive, a patterned layer of adhesive or any array of separate lines, spirals or spots of adhesive. Alternatively, the bonding means can comprise heat bonds, pressure bonds, ultrasonic bonds, dynamic mechanical bonds or any other suitable bonding means or combinations of these bonding means as are known in the art. Bonding can be around the perimeter of the cleaning pad (e.g., heat sealing the scrubbing layer and optional attachment layer and/or scrim material), and/or across the area (i.e., the X-Y plane) of the cleaning pad so as to form a pattern on the surface of the cleaning pad. Bonding the layers of the cleaning pad with ultrasonic heating to form bonds across the area of the pad will provide integrity to avoid shearing of the discrete pad layers during use.

Referring to the figures which depict the cleaning pad of the present invention, Figure 3 is a perspective view of a removable cleaning pad 200 comprising a scrubbing layer 201, an attachment layer 203 and an absorbent layer 205 positioned between the scrubbing layer and the attachment layer. Cleaning pad 200 is not depicted as having multiple substantially planar surfaces. As indicated above, while Figure 3 depicts each

of layers 201, 203 and 205 as a single layer of material, one or more of these layers can consist of a laminate of two or more plies. For example, in a preferred embodiment, scrubbing layer 201 is a two-ply laminate of carded polypropylene, where the lower layer is slitted. Also, though not depicted in Figure 3, materials that do not inhibit fluid flow can be positioned between scrubbing layer 201 and absorbent layer 203 and/or between absorbent layer 203 and attachment layer 205. However, it is important that the scrubbing and absorbent layers be in substantial fluid communication, to provide the requisite absorbency of the cleaning pad. While Figure 3 depicts pad 200 as having all of the pad's layers of equal size in the X and Y dimensions, it is preferred that the scrubbing layer 201 and attachment layer 205 be larger than the absorbent layer, such that layers 201 and 205 can be bonded together around the periphery of the pad to provide integrity. The scrubbing and attachment layers can be bonded to the absorbent layer or to each other by any of a variety of bonding means, including the use of a uniform continuous layer of adhesive, a patterned layer of adhesive or any array of separate lines, spirals or spots of adhesive. Alternatively, the bonding means can comprise heat bonds, pressure bonds, ultrasonic bonds, dynamic mechanical bonds or any other suitable bonding means or combinations of these bonding means as are known in the art. Bonding can be around the perimeter of the cleaning pad, and/or across the surface of the cleaning pad so as to form a pattern on the surface of the scrubbing layer 201.

Figure 4 is a blown perspective view of the absorbent layer 305 of an embodiment of a cleaning pad of the present invention. The cleaning pad's scrubbing layer and optional attachment layer are not shown in Figure 4. Absorbent layer 305 is depicted in this embodiment as consisting of a tri-laminate structure. Specifically absorbent layer 305 is shown to consist of a discrete layer of particulate superabsorbent gelling material, shown as 307, positioned between two discrete layers 306 and 308 of fibrous material. In this embodiment, because of the region 307 of high concentration of superabsorbent gelling material, it is preferred that the superabsorbent material not exhibit gel blocking discussed above. In a particularly preferred embodiment, fibrous layers 306 and 308 will each be a thermally bonded fibrous substrate of cellulosic fibers, and lower fibrous layer 308 will be in direct fluid communication with the scrubbing layer (not shown). (Layer 307 can alternatively be a mixture of fibrous material and superabsorbent material, where the superabsorbent material is preferably present in a relatively high percentage by weight of the layer.) Also, while depicted as having equal widths, in a preferred embodiment layer 306 will be wider than layer 307 and layer 307 will be wider than layer 308. When a scrubbing and attachment layer are included, such

a combination will provide a pad having the multiple substantially planar surfaces of the present invention.

Figure 5 is a cross-sectional view (taken along the y-z plane) of cleaning pad 400 having a scrubbing layer 401, an attachment layer 403, and an absorbent layer indicated generally as 404 positioned between the scrubbing and attachment layers. Absorbent layer 404 consists of three separate layers 405, 407 and 409. Layer 409 is wider than layer 407 which is wider than layer 405. Again, this tapering of absorbent layer materials provides multiple planar surfaces indicated generally as 411, 413 and 415. (For purposes of discussion, surface 411 is referred to as the front edge of the cleaning pad 400 when the pad is attached to an implement; surface 413 is referred to as the back edge of pad 400.) In one embodiment, layers 405 and 407 comprise a high concentration of superabsorbent material, while layer 409 contains little or no superabsorbent material. In such embodiments, one or both of layers 405 and 407 can be comprised of a homogenous blend of superabsorbent material and fibrous material. Alternatively, one or both layers can be comprised of discrete layers, e.g., two fibrous layers surrounding an essentially continuous layer of superabsorbent particles.

Though not a requirement, Applicants have found that it can be desirable to reduce the level of or eliminate superabsorbent particles at the extreme front and rear edges. This is accomplished in pad 400 by constructing absorbent layer 409 without superabsorbent material.

V. Test Methods

A. Performance Under Pressure

This test determines the gram/gram absorption of deionized water for a cleaning pad that is laterally confined in a piston/cylinder assembly under an initial confining pressure of 0.09 psi (about 0.6 kPa). (Depending on the composition of the cleaning pad sample, the confining pressure can decrease slightly as the sample absorbs water and swells during the time of the test.) The objective of the test is to assess the ability of a cleaning pad to absorb fluid, over a practical period of time, when the pad is exposed to usage conditions (horizontal wicking and pressures).

The test fluid for the PUP capacity test is deionized water. This fluid is absorbed by the cleaning pad under demand absorption conditions at near-zero hydrostatic pressure.

A suitable apparatus 510 for this test is shown in Figure 5. At one end of this apparatus is a fluid reservoir 512 (such as a petri dish) having a cover 514. Reservoir 512 rests on an analytical balance indicated generally as 516. The other end of apparatus 510 is a fritted funnel indicated generally as 518, a piston/cylinder assembly indicated

generally as 520 that fits inside funnel 518, and cylindrical plastic fritted funnel cover indicated generally as 522 that fits over funnel 518 and is open at the bottom and closed at the top, the top having a pinhole. Apparatus 510 has a system for conveying fluid in either direction that consists of sections glass capillary tubing indicated as 524 and 531a, flexible plastic tubing (e.g., 1/4 inch i.d. and 3/8 inch o.d. Tygon tubing) indicated as 531b, stopcock assemblies 526 and 538 and Teflon connectors 548, 550 and 552 to connect glass tubing 524 and 531a and stopcock assemblies 526 and 538. Stopcock assembly 526 consists of a 3-way valve 528, glass capillary tubing 530 and 534 in the main fluid system, and a section of glass capillary tubing 532 for replenishing reservoir 512 and forward flushing the fritted disc in fritted funnel 518. Stopcock assembly 538 similarly consists of a 3-way valve 540, glass capillary tubing 542 and 546 in the main fluid line, and a section of glass capillary tubing 544 that acts as a drain for the system.

Referring to Figure 6, assembly 520 consists of a cylinder 554, a cup-like piston indicated by 556 and a weight 558 that fits inside piston 556. Attached to bottom end of cylinder 554 is a No. 400 mesh stainless steel cloth screen 559 that is biaxially stretched to tautness prior to attachment. The cleaning pad sample indicated generally as 560 rests on screen 559 with the surface-contacting (or scrubbing) layer in contact with screen 559. The cleaning pad sample is a circular sample having a diameter of 5.4 cm. (While sample 560 is depicted as a single layer, the sample will actually consist of a circular sample having all layers contained by the pad from which the sample is cut.) Cylinder 554 is bored from a transparent LEXAN[®] rod (or equivalent) and has an inner diameter of 6.00 cm (area = 28.25 cm²), with a wall thickness of approximately 5 mm and a height of approximately 5 cm. The piston 556 is in the form of a Teflon cup and is machined to fit into cylinder 554 within tight tolerances. Cylindrical stainless steel weight 558 is machined to fit snugly within piston 556 and is fitted with a handle on the top (not shown) for ease in removing. The combined weight of piston 556 and weight 558 is 145.3 g, which corresponds to a pressure of 0.09 psi for an area of 22.9 cm².

The components of apparatus 510 are sized such that the flow rate of deionized water therethrough, under a 10 cm hydrostatic head, is at least 0.01 g/cm²/sec, where the flow rate is normalized by the area of fritted funnel 518. Factors particularly impactful on flow rate are the permeability of the fritted disc in fritted funnel 518 and the inner diameters of glass tubing 524, 530, 534, 542, 546 and 531a, and stopcock valves 528 and 540.

Reservoir 512 is positioned on an analytical balance 516 that is accurate to at least 0.01g with a drift of less than 0.1g/hr. The balance is preferably interfaced to a computer with software that can (i) monitor balance weight change at pre-set time

intervals from the initiation of the PUP test and (ii) be set to auto initiate on a weight change of 0.01-0.05 g, depending on balance sensitivity. Capillary tubing 524 entering the reservoir 512 should not contact either the bottom thereof or cover 514. The volume of fluid (not shown) in reservoir 512 should be sufficient such that air is not drawn into capillary tubing 524 during the measurement. The fluid level in reservoir 512, at the initiation of the measurement, should be approximately 2 mm below the top surface of fritted disc in fritted funnel 518. This can be confirmed by placing a small drop of fluid on the fritted disc and gravimetrically monitoring its slow flow back into reservoir 512. This level should not change significantly when piston/cylinder assembly 520 is positioned within funnel 518. The reservoir should have a sufficiently large diameter (e.g., ~14 cm) so that withdrawal of ~40 ml portions results in a change in the fluid height of less than 3 mm.

Prior to measurement, the assembly is filled with deionized water. The fritted disc in fritted funnel 518 is forward flushed so that it is filled with fresh deionized water. To the extent possible, air bubbles are removed from the bottom surface of the fritted disc and the system that connects the funnel to the reservoir. The following procedures are carried out by sequential operation of the 3-way stopcocks:

1. Excess fluid on the upper surface of the fritted disc is removed (e.g. poured) from fritted funnel 518.
2. The solution height/weight of reservoir 512 is adjusted to the proper level/value.
3. Fritted funnel 518 is positioned at the correct height relative to reservoir 512.
4. Fritted funnel 518 is then covered with fritted funnel cover 522.
5. The reservoir 512 and fritted funnel 518 are equilibrated with valves 528 and 540 of stopcock assemblies 526 and 538 in the open connecting position.
6. Valves 528 and 540 are then closed.
7. Valve 540 is then turned so that the funnel is open to the drain tube 544.
8. The system is allowed to equilibrate in this position for 5 minutes.
9. Valve 540 is then returned to its closed position.

Steps Nos. 7-9 temporarily "dry" the surface of fritted funnel 518 by exposing it to a small hydrostatic suction of ~5 cm. This suction is applied if the open end of tube 544 extends ~5 cm below the level of the fritted disc in fritted funnel 518 and is filled with deionized water. Typically ~0.04 g of fluid is drained from the system during this procedure. This procedure prevents premature absorption of deionized water when

piston/cylinder assembly 520 is positioned within fritted funnel 518. The quantity of fluid that drains from the fritted funnel in this procedure (referred to as the fritted funnel correction weight, or "Wffc") is measured by conducting the PUP test (see below) for a time period of 20 minutes without piston/cylinder assembly 520. Essentially all of the fluid drained from the fritted funnel by this procedure is very quickly reabsorbed by the funnel when the test is initiated. Thus, it is necessary to subtract this correction weight from weights of fluid removed from the reservoir during the PUP test (see below).

A round die-cut sample 560 is placed in cylinder 554. The piston 556 is slid into cylinder 554 and positioned on top of the cleaning pad sample 560. The piston/cylinder assembly 520 is placed on top of the frit portion of funnel 518, the weight 558 is slipped into piston 556, and the top of funnel 518 is then covered with fritted funnel cover 522. After the balance reading is checked for stability, the test is initiated by opening valves 528 and 540 so as to connect funnel 518 and reservoir 512. With auto initiation, data collection commences immediately, as funnel 518 begins to reabsorb fluid.

Data is recorded at intervals over a total time period of 1200 seconds (20 minutes). PUP absorbent capacity is determined as follows:

$$t_{1200} \text{ absorbent capacity (g/g)} = [W_{r(t=0)} - W_{r(t=1200)} - W_{ffc}] / W_{ds}$$

where t_{1200} absorbent capacity is the g/g capacity of the pad after 1200 seconds, $W_{r(t=0)}$ is the weight in grams of reservoir 512 prior to initiation, $W_{r(t=1200)}$ is the weight in grams of reservoir 512 at 1200 seconds after initiation, W_{ffc} is the fritted funnel correction weight and W_{ds} is the dry weight of the cleaning pad sample. It follows that the sample's t_{30} and t_{900} absorbent capacities are measured similarly, except $W_{r(t=30)}$ and $W_{r(t=900)}$ (i.e., the weight of the reservoir at 30 seconds and 900 seconds after initiation, respectively) are used in the above formula. The t_{30} percent absorbency of the sample is calculated as $[t_{30} \text{ absorbent capacity}] / [t_{1200} \text{ absorbent capacity}] \times 100\%$.

B. Squeeze-out

The ability of the cleaning pad to retain fluid when exposed to in-use pressures, and therefor to avoid fluid "squeeze-out", is another important parameter to the present invention. "Squeeze-out" is measured on an entire cleaning pad by determining the amount of fluid that can be blotted from the sample with Whatman filter paper under pressures of 0.25 psi (1.5 kPa). Squeeze-out is performed on a sample that has been saturated to capacity with deionized water via horizontal wicking (specifically, via wicking from the surface of the pad consisting of the scrubbing or surface-contacting layer). (One means for obtaining a saturated sample is described as the Horizontal

Gravimetric Wicking method of U.S. application Serial No. 08/542,497 (Dyer et al.), filed October 13, 1995, which is incorporated by reference herein.) The fluid-containing sample is placed horizontally in an apparatus capable of supplying the respective pressures, preferably by using an air-filled bag that will provide evenly distributed pressure across the surface of the sample. The squeeze-out value is reported as the weight of test fluid lost per weight of the wet sample.

EXAMPLES

Context of Use with Absorbent Cleaning pad

Detergent composition/solution containing about 0.12% of detergent surfactant, comprising a linear alcohol ethoxylate detergent surfactant (Neodol 1-5[®], available from Shell Chemical Co.) and an alkylsulfonate (Bioterge[®] PAS-8s, a linear C₈ sulfonate available from Stepan Co.); about 1%, ethanol (Quantum Chemicals), about 0.75% propylene glycol n-butyl ether (Dow Co.); about 0.006% Dow Corning AF suds suppressor (Dow) and about 0.05% 2-amino-2-methyl-1-propanol; adjuvants including dyes and perfumes; and the balance deionized water is used as a base in which various polymers and gums are added for performance comparisons in floor end result cleaning. This testing is done in the context of an absorbent cleaning pad (containing an effective amount of sodium polyacrylate, preferably cross-linked sodium polyacrylate, a superabsorbent material).

TEST PROTOCOL

Testing involves soiling a 2'X2' floor area each with about 8.0 ml of an oily particulate soil solution using a paint roller (about 0.5 g soil applied to tile after solvent evaporated). Each floor area is then cleaned using 8 ml of solution (applied to bottom 2 tiles) and an absorbent pad (disclosed within this filing) of the following dimension approximately 5.75" x 5.75". The cleaning pad is attached to a velcro mop head on a handle and wiped across the floor surface using an up-and-down motion, going over the surface one way and then back the other way. Floors are then graded for end result cleaning appearance at different time intervals (about 10, 30, and 60 minutes). The tiles are then re-soiled and a second cleaning test is run. In the second test, the same soiled pads from the first test are used to simulate stress cleaning situation and determine the effect of build-up. The End Result Comparison is based upon a 0-4 Scale where 0 is none and 4 is severe streaks. The following are examples of some of the data (grade difference of 0.25 is significant):

Table 1	Example 1	Example 2	Example 3	Example 4	Example 5	Example 6	Example 7
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End Result Comp- arison	No polymer	0.005% Xanthan gum	0.015% Xanthan Gum	0.15% Polyvinyl Pyrolidone/ Acrylic Acid	0.05% Polystyrene Sulfonate	0.008% Polyvinyl Pyrolidone	0.005% Xanthan Gum + 0.008% Polyvinyl Pyrolidone
Test 1	1.25	0.5	0.75	-	-	-	-
Test 2	-	0.5	-	1.75	1.25	-	-
Test 3	1.25	0.5	-	-	-	1.25	-
Test 4	1.25	0.5	-	-	-	-	0.75

Table 2	Example 1	Example 2	Example 8	Example 9	Example 10	Example 11	Example 12	Example 13
End Result Comparison	No polymer	0.005 Xanthan gum	0.08 Hydroxy propyl non-ionic gum Jaguar N HP120	0.08 Hydroxy propyl cationic gum Jaguar C-17	0.08 Gum Arabic	0.08 Pectin	0.005 Poly acrylate	0.005 Xanthan Gum + 0.005 Poly acrylate
Test 5	1.25	0.75	-	-	-	-	1.5	-
Test 6	1.25	0.5	-	-	-	-	-	1.0
Test 7	-	0.5	1.0	0.75	-	-	-	-
Test 8	1.75	0.75	-	-	1.25	-	-	-
Test 9	1.75	0.75	-	-	-	1.0	-	-

The data in the Table 1 and 2 clearly shows the benefit of using specific polymers especially at low levels. Xanthan gum, Jaguar[®] polymers, Pectin and Gum Arabic are particularly impressive. Also comparing examples 6 vs 7 and example 12 vs 13 shows how Xanthan gum can work synergistically with other polymers to provide an improvement.

The impact of polymers added to conventional cleaners diluted according to recommended dilution (using distilled water) but used in the context of absorbent cleaning pad were tested using the protocol listed above. The indicated polymers and levels are used in Mr. Clean[®], and Pinesol[®] (lemon), commercially available products. Results are as follows:

Table 3	Example 2	Example 14	Example 15	Example 16	Example 17
End Result Comparison	Solution Base from above + 0.005 Xanthan gum	Mr. Clean All-purpose 0.75% dilution	Mr. Clean All-purpose 0.75% dilution + 0.005 Xanthan Gum	Pinesol Lemon 1.5% dilution	Pinesol Lemon 1.5% dilution + 0.005 Xanthan Gum
Test 10	0.5	1.75	1.0	-	-
Test 11	0.75	-	-	2.0	1.5

The data in Table 3 clearly shows that xanthan gum can improve the end result of conventional floor cleaners when diluted to recommended dilution and used in the context of the absorbent cleaning pad (disclosed in this filing).

Conventional Mop Testing

To further dimensionalize the benefit of hydrophilic polymers in the context of a conventional cleaner using conventional cleaning implements, testing is done using the

soiling protocol for the previous testing. The implements used and application protocol are different as follows:

Sponge Mop Simulation:

An approximately 2.5" X 3.5" X 1" sponge is attached to handle soaked in the appropriate solution and wrung to dampness (about 60 ml solution absorbed in dry sponge). The sponge is then wiped across the soiled floor surface in an up-and-down motion passing over the surface once than back in the other direction.. Floors are then graded for end result appearance after completely drying using 0-4 scale (0=best and 4=worst).

Strip Mop Simulation:

A Libman strip mop head is taken and strips are cut down to 4.75" lengths to form a mini strip mop. The mini strip mop head is then soaked in the appropriate solution and wrung to dampness (about 130 g solution absorbed in dry implement). Each mini strip mop head is then wiped across the soiled floor surface in side-to-side motion, passing over the entire surface. The mini strip mop is then passed in an up-and-down motion across the entire surface to simulate a wipe pattern used by consumers when using a strip mop. The floors are then graded for end result appearance after completely drying using the 0 - 4 scale.

Floor Cloth Simulation:

A European floor cloth (referred to as a Serpien) is cut to an approximately 9"X10" dimension. The floor cloth is then soaked in the appropriate solution and wrung to dampness about 70 g solution absorbed in dry implement). Using an approximately 5"X 5" flat mop head attached to a handle the floor cloth is wiped across soiled floor surface in an up-and-down motion passing over surface once than back in the other direction.. Floors are then graded for end result appearance after completely drying using the 0-4 scale.

Commercially available conventional products Mr. Clean®, and Pinesol® (lemon) solutions are diluted according to recommended dilution instructions (using approximately 7 g tap water). These solutions are then tested using the conventional mops with and without xanthan gum.

Table 4	Example 18	Example 19	Example 20	Example 21	Example 22	Example 23
End Result Comparison	Sponge Mop with Mr. Clean All-purpose 0.75% dilution	Sponge Mop with Mr. Clean All-purpose 0.75% dilution + 0.005 Xanthan Gum	Strip Mop with Mr. Clean All-purpose 0.75% dilution	Strip Mop with Mr. Clean All-purpose 0.75% dilution + 0.005 Xanthan Gum	Floor Cloth with Mr. Clean All-purpose 0.75% dilution	Floor Cloth with Mr. Clean All-purpose 0.75% dilution + 0.005 Xanthan Gum
Test 12	3.0	1.75	-	-	-	-
Test 13	-	-	1.5	1.0	-	-
Test 14	-	-	-	-	2.0	1.5

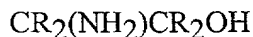
Table 5	Example 24	Example 25	Example 26	Example 27
End Result Comparison	Sponge Mop with Pinesol Lemon 1.5% dilution	Sponge Mop with Pinesol Lemon 1.5% dilution + 0.005 Xanthan Gum	Strip Mop with Pinesol Lemon 1.5% dilution	Strip Mop with Pinesol Lemon 1.5% dilution + 0.005 Xanthan Gum
Test 15	3.0	2.5	-	-
Test 16	-	-	2.25	1.75

The data in Tables 4 and 5 again shows that xanthan gum can even improve the end result of commercially available conventional floor cleaners when diluted using recommended instructions and used in the context of conventional mopping systems.

WHAT IS CLAIMED IS:

1. Detergent composition for cleaning floors comprising either: (1) from about 0.0001% to about 0.2%, (2) from about 0.0001% to about 0.1%, or (3) from about 0.0005% to about 0.08%, by weight of the composition, of hydrophilic, shear-thinning polymer that is capable of inhibiting molecular aggregation of surfactant solution on floors during the dry-down process.
2. The detergent composition according to Claim 1 wherein the hydrophilic, shear-thinning polymer either has a molecular weight of at least about 100,000 or a molecular weight of at least about 1,000,000, and optionally is xanthan gum, or, also optionally, is selected from the group consisting of xanthan gum, guar gum, gum arabic, pectin and mixtures thereof.
3. The detergent composition of Claim 1 or Claim 2 wherein either: (1) the level of detergent surfactant is from about 0.01% to about 0.50%; the level of one or more solvents is less than about 5.0%; and the pH is greater than about 9; (2) the level of detergent surfactant is from about 0.05% to about 0.30%; the level of one or more solvents is less than about 5.0%; and the pH is greater than about 9.5; or (3) the level of detergent surfactant is from about 0.10% to about 0.20%; the level of one or more solvents is less than about 5.0%; and the pH is greater than about 10.0.
4. A detergent composition according to any of Claims 1-3 wherein the composition is obtained by dilution with from about 1:50 to about 1:250 parts per part of the composition of water of a more concentrated product form prior to use.
5. The detergent composition of any of Claims 1-4 containing an effective amount of suds suppressor, said effective level optionally being either a level of from about 0.0005% to about 0.02% or a level of from about 0.001% to about 0.01%, and said suds suppressor optionally comprising a silicone suds suppressor.
6. The detergent composition of Claim 1 wherein the detergent surfactant has a predominantly linear structure and optionally is selected from the group consisting of straight chain anionic and nonionic detergent surfactants.

7. The detergent composition of Claim 1 wherein the alkalinity is provided, at least in an effective amount, by volatile alkaline agent, said volatile alkaline agent optionally being an alkanol amine having the formula:



wherein each R is selected from the group consisting of hydrogen and alkyl groups containing from one to four carbon atoms and the total of carbon atoms in the compound is from three to six, which optionally is 2-amino-2-methyl-1-propanol.

8. A kit comprising an implement containing a pad containing superabsorbent material and a detergent composition containing no more than about 0.5% detergent surfactant; a level of one, or more solvents, that is less than about 5.0%; and a pH of more than about 9.

9. The detergent composition of any of Claims 1-7 in a container, in association with instructions to use it with a pad containing a superabsorbent material.

10. The process of cleaning a surface, optionally comprising ceramic, vinyl, polyurethane coated vinyl, wood, pre-finished wood, and/or wood laminate surfaces, comprising applying an effective amount of a detergent composition containing no more than about 0.5% detergent surfactant; a level of one or more solvents, that is less than about 5.0%; optionally an effective amount of suds suppressor, and having a pH of more than about 9 and absorbing the composition in an absorbent structure comprising a superabsorbent material, or, optionally, absorbing the composition using a string, sponge, strip mop or floor cloth.

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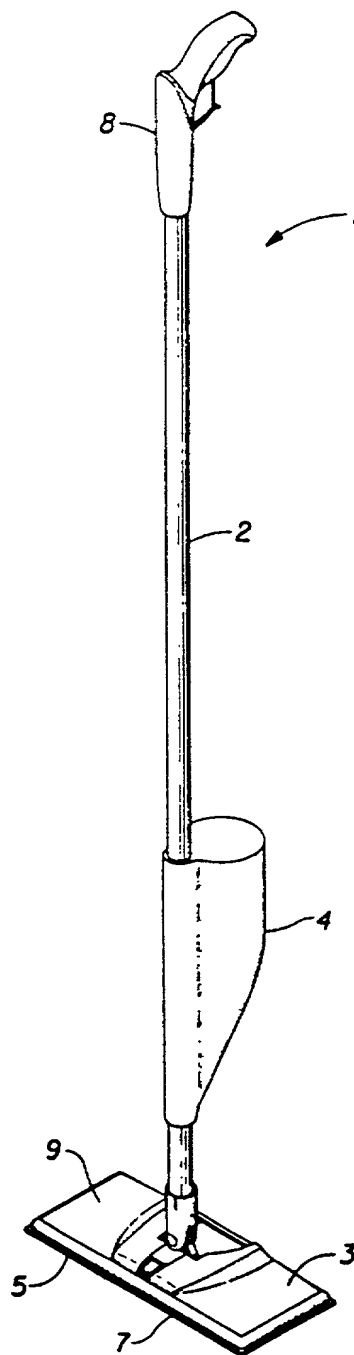


Fig. 1a

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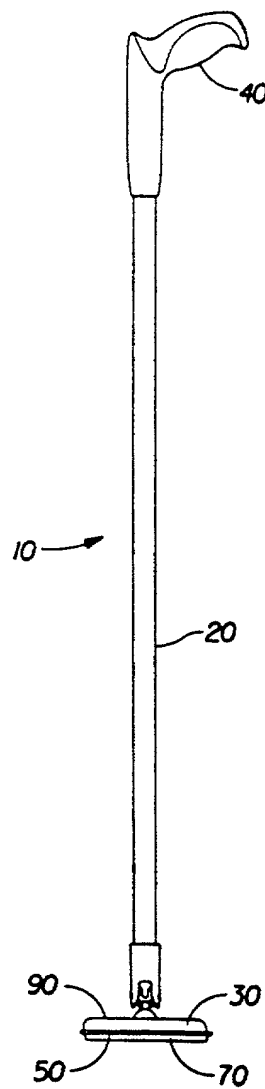


Fig. 1b

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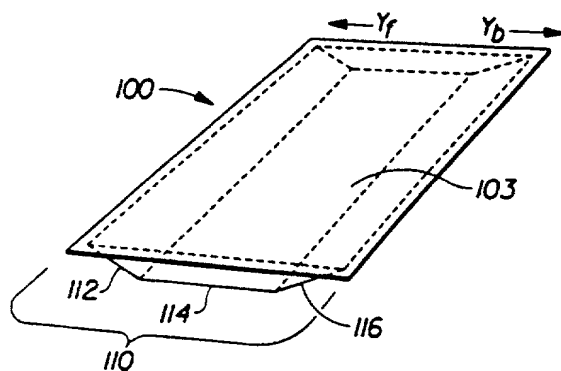


Fig. 2

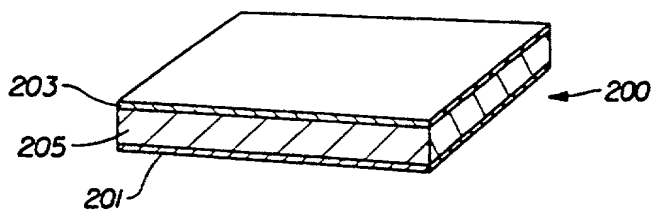


Fig. 3

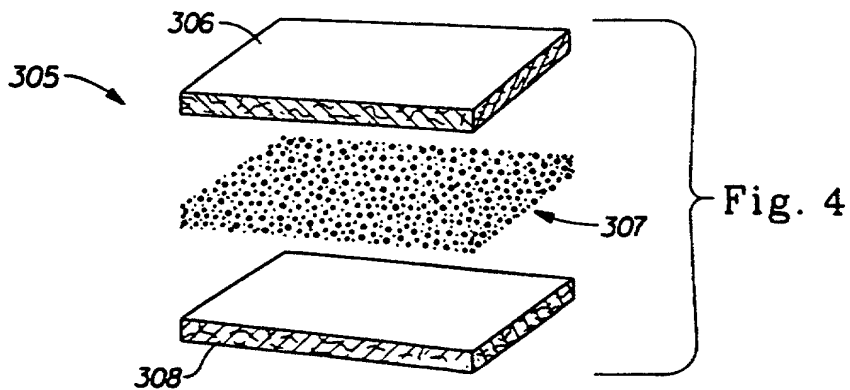


Fig. 4

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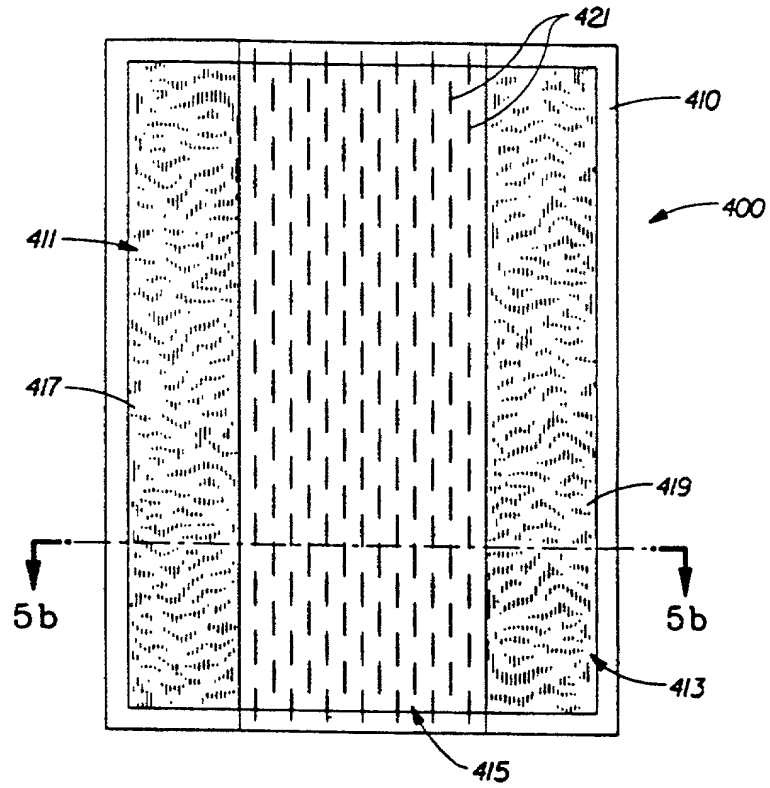


Fig. 5a

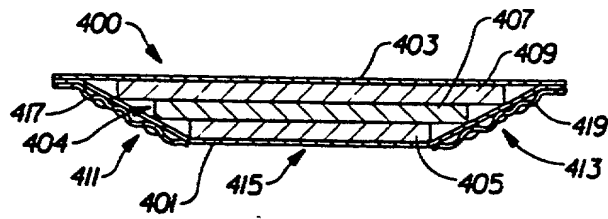


Fig. 5b

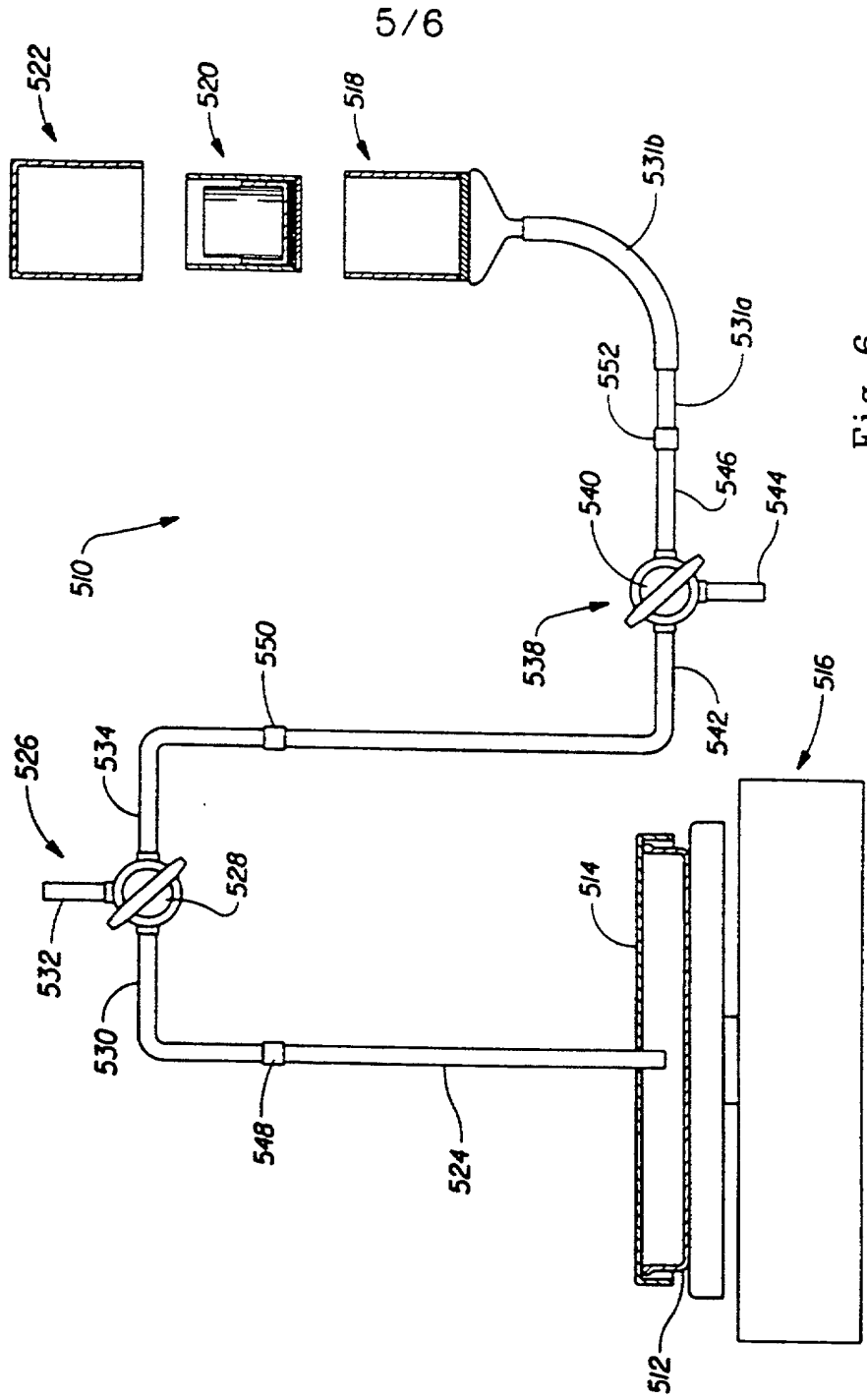


Fig. 6

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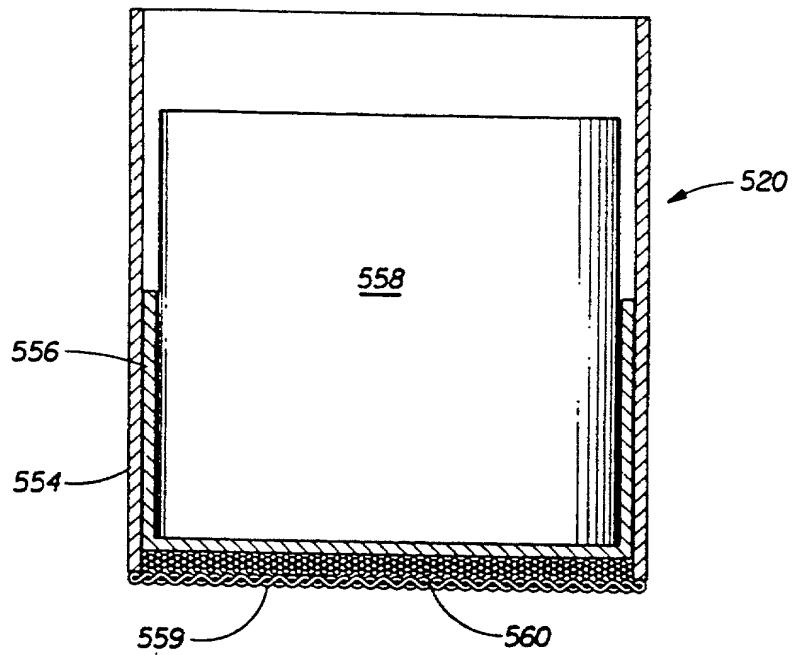


Fig. 7

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled

DETERGENT COMPOSITION FOR HARD SURFACES COMPRISING HYDROPHILIC SHEAR-THINNING POLYMER AT VERY LOW LEVEL

the specification of which

(check ☐ is attached hereto.
one) ☒ was filed on October 1, 1998 as
Application Serial No. PCT/US98/20513
and was amended on _____

(if applicable)

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to the examination of this application in accordance with Title 37 Code of Federal Regulations §1.56(a).

I hereby claim foreign priority benefits under Title 35 United States Code §119(a)-(d) of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed:

Prior Foreign Application(s)

Priority Claimed

_____ (Number)	_____ (Country)	_____ (Day/Month/Year Filed)	<input type="checkbox"/> Yes	<input type="checkbox"/> No
_____ (Number)	_____ (Country)	_____ (Day/Month/Year Filed)	<input type="checkbox"/> Yes	<input type="checkbox"/> No

I hereby claim the benefit under Title 35, United States Code §119(e) of any United States provisional application(s) listed below.

<u>60/086,447</u>	<u>5/22/98</u>	<u>60/061,296</u>	<u>10/7/97</u>
Application Serial No.	Filing Date	Application Serial No.	Filing Date

I hereby claim the benefit under Title 35 United States Code §120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35 United States Code §112, I acknowledge the duty to disclose material information as defined in Title 37 Code of Federal Regulations §1.56(a) which occurred between the filing date of the prior application and the national or PCT international filing date of this application:

_____ (Application Serial No.)	_____ (Filing Date)	_____ (Status) (patented, pending, abandoned)
_____ (Application Serial No.)	_____ (Filing Date)	_____ (Status) (patented, pending, abandoned)

I hereby appoint the following as my attorney(s) or agent(s) with full power of substitution to prosecute this application and transact all business in the Patent and Trademark Office connected therewith:

<u>Atty Name</u>	<u>Atty Reg Number</u>	<u>Associate Power of Attorney Attached</u>
Robert B. Aylor	<u>20,369</u>	
Brian M. Bolam	<u>37,513</u>	
Jacobus C. Rasser	<u>37,043</u>	<input checked="" type="checkbox"/> No
T. David Reed	<u>32,931</u>	
Kim W. Zerby	<u>32,323</u>	
John M. Pollaro	<u>27,670</u>	

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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In the matter of

U. S. National Phase Entry :
Under 35 USC 371 from :
the International Application of :
POLICICCHIO, Nicola John et al :
Int'l Application No. PCT/US98/20513 :
Filed in the RO/US on 01 October 1998 :
Entitled: Detergent Composition For Hard :
Surfaces Comprising Hydrophilic Shear- :
Thinning Polymer at Very Low Level :

ASSOCIATE POWER OF ATTORNEY

Assistant Commissioner for Patents
Box PCT
Washington, D.C. 20231

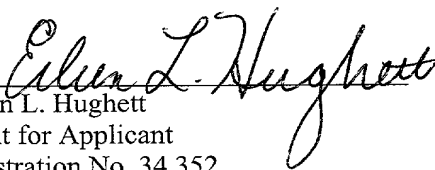
Dear Sir:

You are requested to recognize J. J. Camp (Registration No. 44,582); K. W. Zerby (Registration No. 32,323); B. M. Bolam (Registration No. 37,513); C. B. Cook (Registration No. 39,151); M. Dressman (Registration No. 42,498); R. S. Echler, Sr. (Registration No. 41,006); D. M. Goodrich (Registration No. 42,592); I. S. Robinson (Registration No. 43,348); and J. C. Vago (Registration No. 40,855) of The Procter & Gamble Company, Cincinnati, Ohio, as Associate Attorney to prosecute this application, to make alterations and amendments therein, and to transact all business in the Patent Office connected with the application or with the patent granted thereupon.

Please address all future communications to:

J. J. Camp
Patent Division
The Procter & Gamble Company
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Cincinnati, Ohio 45241

Respectfully submitted for Applicants,

By 
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06 April 2000
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I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

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